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The Journal
of the
SOCIETY OF DYERS AND COLOURISTS

Volume 70 Number 7

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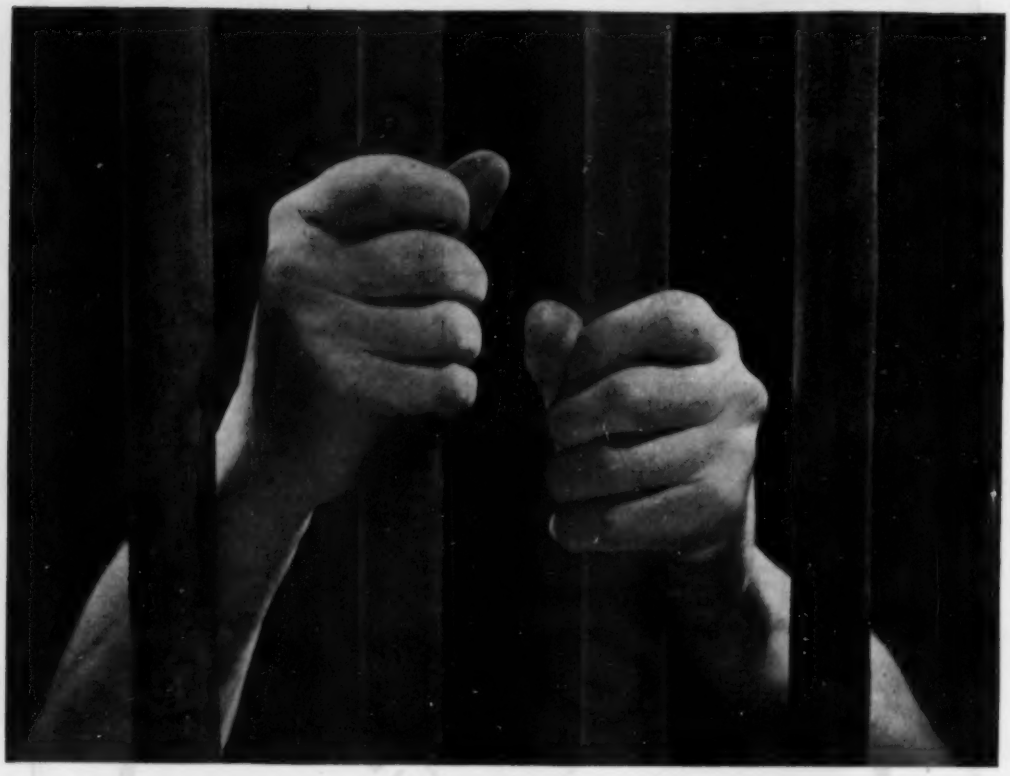

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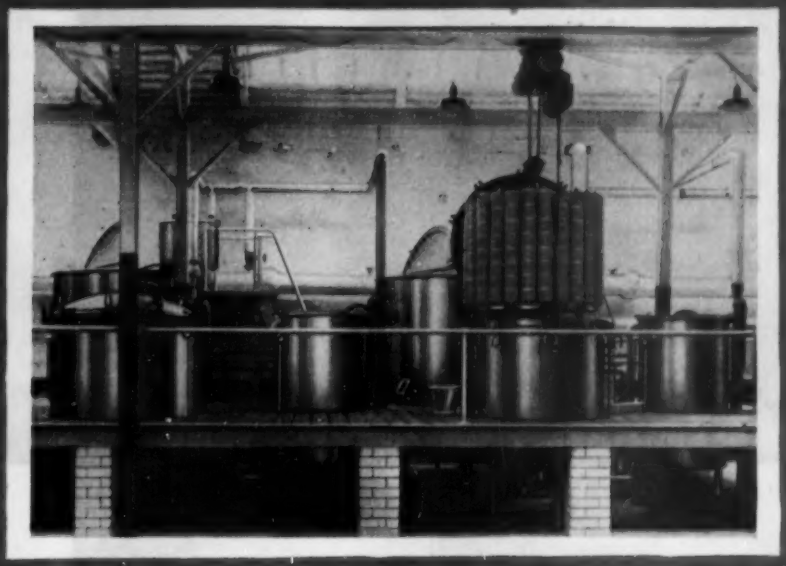

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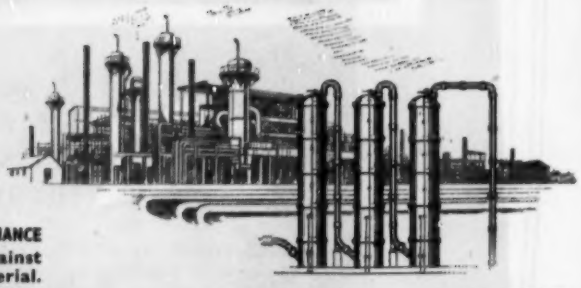

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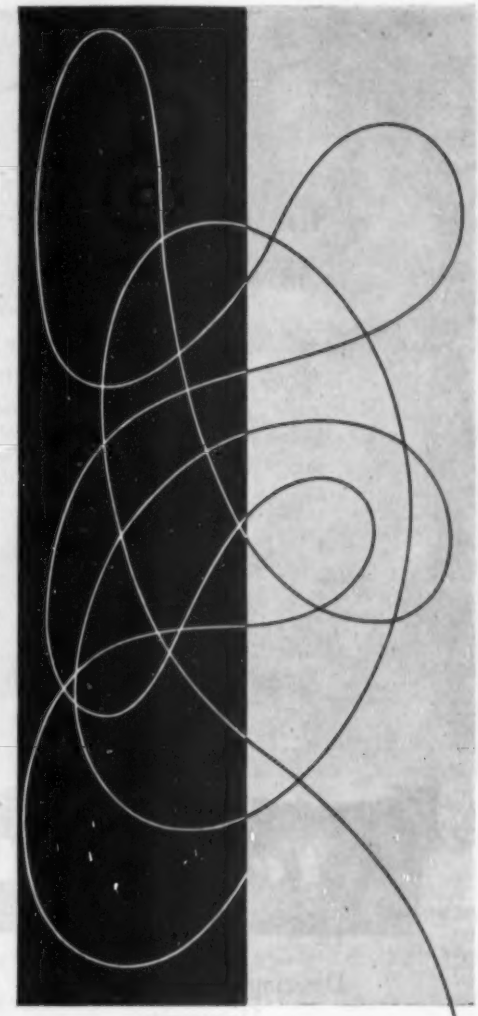

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Forthcoming Papers

The following papers have been accepted by the Publications Committee, and will appear in future issues of the *Journal*—

LECTURES

Level Dyeing with Afterchrome Dyes *I. D. Rattee*

Physics in the Dyeing, Printing, and Finishing Industries *N. W. Yelland*

COMMUNICATIONS

The Polarography of Azo Dyes *J. de O. Cabral and H. A. Turner*

Dyeing of Cellulose Acetate with Non-ionic Dyes.
I—Absolute Heats of Association
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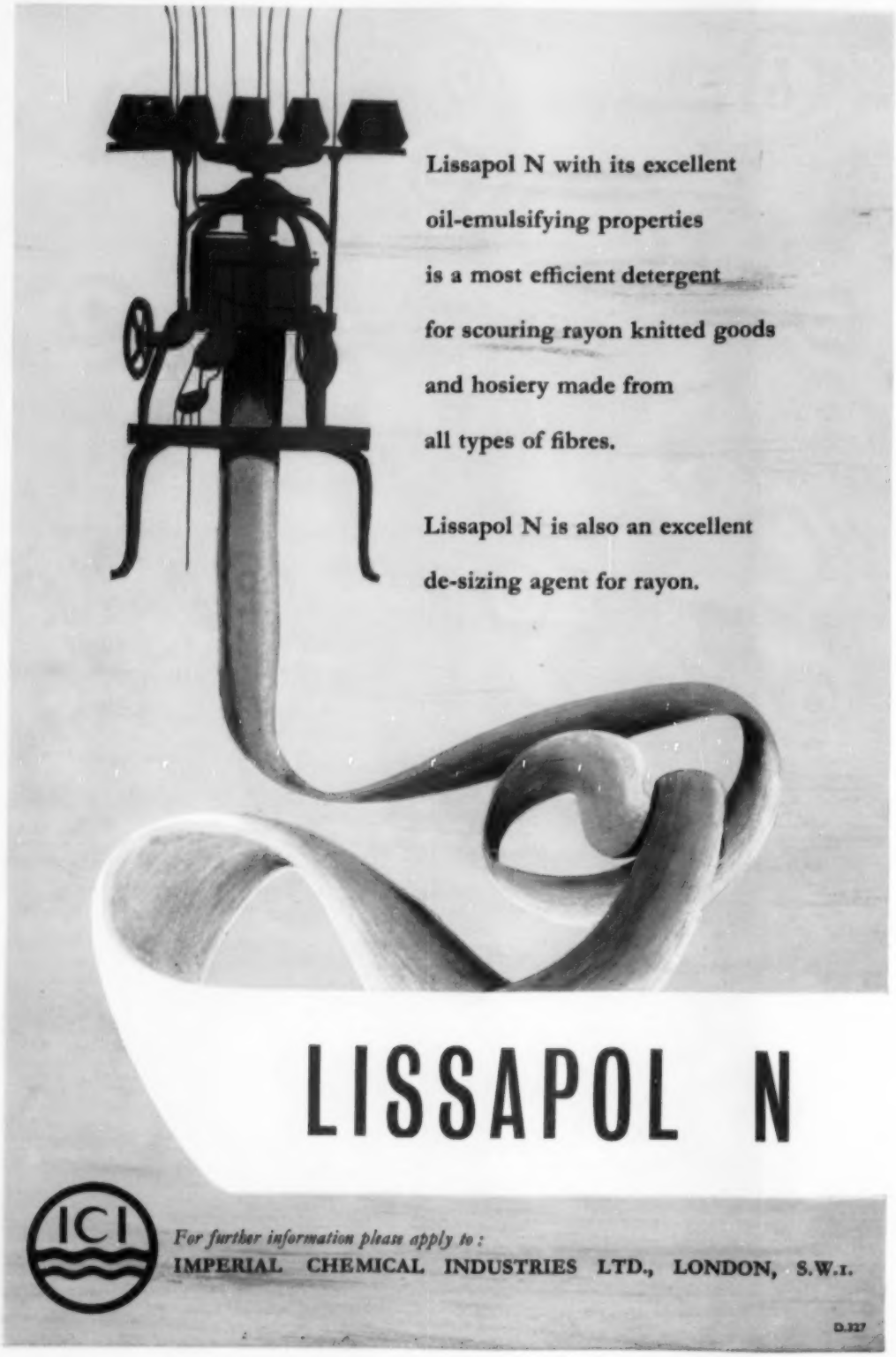
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Volume 70 Number 7 JULY 1954 *Issued Monthly*

It is with very deep sorrow that we have to report the sudden death of our President Frederic Leathley Goodall M.Sc. F.R.I.C. F.S.D.C. on Sunday, 27th June 1954, at his home in Wilmslow, Cheshire.

This is a grievous blow to the Society, but the first thoughts of Members will be with his widow and two daughters in their sad bereavement.

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Presidential Message

IT has been the practice for many years for the President to deliver himself of observations as regards the general health of the Society either at the Annual General Meeting or more usually at the Annual Dinner. It is partly with the object of shortening the proceedings, especially at the Dinner, which should be an occasion when members can meet as many old friends as possible, that I should like to use this means of telling members what I feel about the Society after one year in office as your President.

In a nutshell, the Society is in very good heart. This impression will be confirmed by a study of Council's report and the annual accounts for 1953, which show respectively an increasing volume of activity and a satisfactory state of the Society's finances.

One of the happiest events of my Presidential year has been the arrangements to collaborate with the Textile Institute in a conference to be held in Scarborough during Whit-week of this year. In my view it is not only possible but essentially desirable for our Society to collaborate with other sister-societies in the provision of any occasions or matter which will serve the interests of the Society, and the proposed conference is a very typical example of what I have in mind.

There have been certain innovations, one of which is the announcement of the Society's scheme for award of Fellowship and Associateship. This is a very important land-mark in the history of the Society and is now well in its stride, it being hoped to hold the first examinations during the present year. Important as it is, it represents only one aspect of the service which the Society wishes to render to its membership as a whole, and to the ideal of advancement of technology which is one of the Society's basic objects and which is of such economic and national importance at the present time.

The fact that the Society continues most actively to serve its members' interests is in my view its principal justification for existence. So long as the Society continues on the path of service I have no doubts at all about its success and certainly feel that there is no need for the dim foreboding expressed in some societies about the future.

Another new activity is the work involved in the holding of a Perkin Centenary which is to be worthy of the landmark in the development of industrial organic chemistry represented by the discovery of mauveine in 1856. In this endeavour Council is supported by the active co-operation of the premier purely scientific societies, and I have little doubt that we shall receive equal support from industrial interests.

Like the new scheme of professional qualifications within the Society, the Perkin Centenary involves additional effort on the part of the Society, but in the case of the Perkin Centenary this effort will reach a climax in 1956, whereas in the case of the Associateship and Fellowship, no climax can readily be foreseen.

There are other minor activities which are additional to the traditional ones on which the Society embarks from year to year, all of which contribute to the wellbeing of the science and technology which the Society intends to foster. Looked at as a whole, however, they involve the Society in a continually increasing amount of work, as a result of which Council has been forced to agree that the load carried by the Society's permanent staff is very heavy, and particularly that of Dr. C. J. W. Hooper is too much for one man to bear. In consequence, comparatively recently Mr J. W. Nicholls has been appointed Secretary to the Society, thus relieving Dr. Hooper of a considerable amount of work and enabling him to concentrate on maintaining the high standard of the Society's oldest and still its principal activity, namely, the *Journal*.

So far as it goes, this is an important step forward, but I feel that it will be insufficient unless the membership of the Society as a whole can by some means or other be brought into greater deployment in furtherance of the Society's activities. Because the Society undertakes more work, more people are required to do that work. This does not necessarily mean an increase in membership, although naturally this would be very welcome, but it does mean that greater advantage must be taken by the Society of the capabilities and willingness to act of a much wider circle of members than has perhaps been the case in the past. I commend this thought to all members, and particularly to Sectional and other Meetings where appointments are made to the large number of Committees responsible for carrying out the work of the Society.

F. L. Goodall

President

The Society of Dyers and Colourists
March 1954

Proceedings of the Society

Annual General Meeting

The Seventieth Annual General Meeting of the Society of Dyers and Colourists was held at the Midland Hotel, Manchester 2, on Friday, 30th April 1954, at 5.0 p.m., the President (Mr. F. L. GOODALL) being in the chair, and about fifty members of the Society being present.

The Secretary (Mr. J. W. NICHOLLS) read the minutes of the Sixty-ninth Annual General Meeting, which were accepted as a correct record on the motion of Mr. H. R. HADFIELD.

REPORT OF THE COUNCIL

The activity of the Society has been maintained during the year ended 31st December 1953, and the records of new members, resignations, and deaths show a net increase in membership of 117.

The loss by death of thirteen members has been reported during the year, including Mr. G. E. Holden, President of the Society 1948–1950, and Mr. L. A. Lantz, Chairman of the Fastness Tests Committee for many years. Council regrets to record the death also of Mr. J. V. Armstrong, Honorary Patent Agent to the Society since 1945.

Council is gratified to record that six more members have now completed fifty years' membership of the Society, viz. Messrs. Arnold Highley, H. Levinstein, F. Scholefield, G. E. Knowles, H. Wilkinson, and H. Yeoman. Dr. Levinstein and Mr. Scholefield are both ex-Presidents of the Society.

Council was very disappointed to learn early in the year that Mr. H. H. Bowen was unable to accept nomination as President for a second year. As a result, Mr. F. L. Goodall, who would have been nominated as President-elect, became President of the Society at the Annual General Meeting held in April 1953, and has already played a vigorous part in initiating new developments in the Society's activities.

Council was greatly perturbed to learn from Mr. J. Barritt that, owing to the increasing pressure of his duties at the Wool Industries Research Association, and of his work on the Second Edition of the *Colour Index*, he would have to relinquish the office of Honorary Secretary of the Society. Mr. Barritt had been a tower of strength to the Society during the past seven years, and his loss would be a great blow to the Society, although it was some consolation that he would continue to play an active part in the work of the Society as Chairman of the Publications Committee. Later in the year Council had great pleasure in conferring Honorary Membership on Mr. Barritt in recognition of his outstanding services to the Society.

In July Council had great pleasure in welcoming as Honorary Secretary Mr. John G. Hopkinson, elder son of a former Honorary Secretary (Mr. G. G. Hopkinson). During his short tenure of office Mr. Hopkinson has already had to deal with several very difficult problems, and his courage and enterprise augur well for the future expansion of the Society's activities. Council greatly appreciates Mr. Hopkinson's willingness to shoulder such heavy responsibility at a comparatively youthful age.

Toward the end of the year Council had under consideration the necessary preliminary steps for the proposed expansion of the Society's activities, especially as the post of Assistant Secretary had become vacant. Council accepted the recommendation of the Finance Committee that the duties undertaken by Dr. C. J. W. Hooper as Editor and General Secretary should be divided as in the past, Dr. Hooper to become Editor and Technical Officer, while the new post of Secretary to the Society would be advertised as vacant.

The Sections and Junior Branches have continued to be very active, and more meetings have been held of special interest to ladies. The first London Lecture, given by Sir Ernest Goodale, on *Ancient and Modern Textile Production in London and the Home Counties* was an outstanding success. This series of lectures has been inaugurated by the London Section as a means of spreading the influence of the Society in the London area.

Early in the year Mr. G. S. J. White, Chairman of the Manchester Section, visited Australia, and conveyed cordial greetings from the Society to the affiliated Society of Dyers and Colourists of Australia. A request for affiliation was received late in the year from the newly formed Dyers and Colourists Association of South Africa, with headquarters in Durban, and Council would like to take this opportunity of wishing the Association vigorous growth and every success.

The ties with sister societies have been strengthened in the course of the year. The Society co-operates closely with the Textile Institute, not only in the production of the *Reviews of Textile Progress*, but also in the field of terms and definitions. A Joint Committee of the Institute and the Society was set up to arrange a conference on *Colour and Textiles*, to be held at Scarborough during Whitsun week 1954. Council is especially gratified at the support shown by other scientific and technical societies in connection with the launching of the new Diploma Scheme and the Perkin Centenary celebrations.

Relations with the American Association of Textile Chemists and Colorists remain cordial, and the two bodies are working hard together both on the production of the Second Edition of the *Colour Index* and on the international standardisation of fastness testing, the latter under the ægis of the International Standards Organisation (I.S.O.).

During September 1953 the International Federation of Associations of Textile Chemists and Colourists held at Lucerne its first post-war Congress, in conjunction with the Annual Conference of the Association des Chimistes de l'Industrie Textile. The Society was represented by Mr. E. A. Swift, whose report to Council described the

success of the function and stressed the importance to the Society of active co-operation with the Continental societies.

FINANCE COMMITTEE— This Committee has met at regular intervals during the year and has kept a close watch on the Society's finances. The Committee has been especially concerned with the prepayments scheme for the Second Edition of the *Colour Index*, and is very gratified at the good response shown in the accounts.

DIPLOMA SCHEME— The Diploma Committee and its Subcommittees have been very active, and Council has considered in great detail the scheme prepared by the Committee. After approval by Council, this was sent for information to several interested sister societies in fields overlapping with our own, and Council is most gratified at the friendly reception given to the Society's plans.

The scheme was finally published in the November issue of the *Journal*, and already several applications have been received for registration as Students and for admission to the examinations for Associate and Fellow of the Society. It is intended to hold the first examination for A.S.D.C. later in 1954. Council is most grateful to the members of the Diploma Committee and its Executive Subcommittee for the time and energy they have put into this important new phase of the Society's activities.

PUBLICATIONS COMMITTEE— The standard of the *Journal* has been maintained during the past year, the number of papers and communications being approximately the same as in 1952. Toward the end of the year, owing to staff and other difficulties, publication of papers became slightly in arrears, and in order to help to meet this situation the Committee, with the approval of Council, took the novel step of publishing in December both a normal issue of the *Journal* and one consisting of the *Proceedings of the Symposium on Textile Printing*. It is expected that the slight delay between receipt of manuscripts and their publication will be reduced to normal proportions early in 1954. Again the Committee is grateful to those firms whose generosity enables reproductions in colour to be included where necessary in scientific papers.

Early in the year Mr. C. L. Bird announced that he felt unable to continue as Chairman of the Publications Committee, and in June the Committee elected Mr. J. Barritt as Chairman. Council wishes to record its high appreciation of the services of Mr. Bird in taking the responsibility for the production of the *Journal* for the past four years, and regrets that he has not been able to continue, but it is very gratifying that in this way Mr. Barritt is maintaining an active interest in the Society's affairs.

Council wishes to record its continued gratitude to all who have contributed to the production of the *Journal*, especially to authors of papers, referees, abstractors, Mr. C. O. Clark as Editor of the abstracts of patents, and Mrs. E. G. Thomson as Indexer.

The third impression of the second edition of *Identification of Dyes on Textile Fibres* by Ellis Clayton and the second edition of *The Theory and Practice of Wool Dyeing* by C. L. Bird have met with a steady sale during the year. The *Review of Textile Progress*, published jointly with the Textile Institute, has now an established position as an annual publication, and a steady demand is shown for all four volumes published so far.

COLOUR INDEX — SECOND EDITION — The members of the *Colour Index* Editorial Panel have been extremely active and have had a very harassing time during the year. The collation of information from dyemakers is proceeding, and much of the manuscript is with the printers, Messrs. Chorley & Pickersgill Ltd. and Messrs. Lund Humphries & Co. Ltd., who have continued to co-operate closely with the Panel. It is still hoped that production will be completed during 1955, as planned.

As already mentioned, the response both to the Society's own prepayments scheme and to that of the A.A.T.C.C. has been very gratifying, and was no doubt stimulated by the vigorous publicity which was undertaken during the first part of the year.

Close and cordial liaison with the *Colour Index* Committees of the A.A.T.C.C. has been maintained during the year.

The work of the Panel has now become very onerous, and Council records its indebtedness to members for their endeavours and for the large proportion of their spare time devoted to this work. Gratitude is due also to the firms and institutions who have encouraged members of their staffs to take part in this work, and also to the dyemakers who have willingly supplied the data to be included in the *Colour Index*.

LIBRARY— Interest in the library was stimulated by the list of books and periodicals sent out to members with the January 1953 issue of the *Journal*, and some 100 separate loans have been made during the course of the year. It should perhaps be stressed that most of the books reviewed and many of the periodicals abstracted in the *Journal* are available for loan to members of the Society.

SYMPOSIUM ON TEXTILE PRINTING— This Symposium was held at St. Annes-on-Sea during September, all meetings and social functions being held under one roof at the Hotel Majestic. The success of previous symposia was maintained, the attendance at the technical sessions being 180–200 in spite of the more limited appeal of the subject. This Symposium was characterised by the attendance of a relatively high proportion of non-members of the Society (about 80) and of visitors from overseas (about 30). The eighteen papers maintained a balance between theoretical and practical approaches to textile printing, and included overseas contributions from the U.S.A., France, Germany, and Switzerland. Preprints were distributed two weeks before the Symposium was due to open, and the full proceedings of the Symposium, including discussion, will be available as a separate bound volume early in 1954. A full programme

of social events was arranged, the chief guest at the Symposium Dinner and Dance being Mr. R. M. Lee, Chairman of the Calico Printers' Association Ltd.

GOLF CHAMPIONSHIP—The President, Mr. F. L. Goodall, has very kindly given a trophy, which was presented to the winner of the golf competition which was held at St. Annes following the Symposium on Textile Printing, on Saturday, 26th September 1953. It is hoped to make this competition an annual event. Council is grateful also to Mr. R. J. Hannay and Mr. J. W. Reidy, who presented prizes for the competition at St. Annes.

J. B. WILKINSON RESEARCH SCHOLARSHIP—It was with great pleasure that Council learned toward the end of the year that Mr. Fred Wilkinson had offered to endow a Research Scholarship to be awarded under the ægis of the Society. Negotiations are in hand concerning the details of the award, and a full announcement will be made when these matters have been settled. Council is most grateful to Mr. Wilkinson for his generosity.

PERKIN CENTENARY—Preliminary arrangements have been made in connection with the celebration by the Society in 1956 of the centenary of the discovery of Mauveine by Sir William H. Perkin. Warm support for the Society's proposals has been expressed by the Royal Society, the Chemical Society, the Royal Institute of Chemistry, and the Society of Chemical Industry, who have nominated distinguished representatives to the Society's Perkin Centenary Committee.

An invitation was received from the A.A.T.C.C. for the Society to arrange for a lecture on Sir William H. Perkin at the Perkin Centennial celebrations to be held in New York in September 1956, and Council is most grateful to Sir Robert Robinson, O.M., a Past President of the Royal Society, for accepting Council's invitation to represent the Society on this occasion.

SOCIETY'S MEDALS ADJUDICATION COMMITTEE—On the recommendation of the Committee, Council has awarded the Gold Medal to Mr. H. A. Turner, the Silver Medal to Mr. A. W. Carpenter, and Bronze Medals to Miss M. Forbes and Mr. H. R. Hadfield. Council extends its warmest congratulations to the recipients. The citations in respect of these awards appear on p. 4 of the January 1954 issue of the *Journal*.

WORSHIPFUL COMPANY OF DYERS' RESEARCH MEDAL—Council thanks the Adjudication Committee for its work in reviewing papers published in the *Journal* from July 1952 to June 1953, as a result of which the Court of the Worshipful Company of Dyers has approved the recommendation that the Company's Gold Research Medal be awarded to Dr. R. H. Peters for his work on the reduction properties of vat dyes.

WORSHIPFUL COMPANY OF FELTMAKERS' RESEARCH MEDAL—Council and the Adjudication Committee again have to report with regret that no paper in this field has been published in the *Journal* during the period under review.

GEORGE DOUGLAS LECTURE—The third George Douglas Lecture, on *The Search for New Dyes in relation to Modern Developments in the Textile Field*, was delivered in the University of Leeds on 27th March 1953 by Dr. T. Holbro of Basle, the chair being taken by the then President, Mr. H. H. Bowen. The lecture was subsequently published in the July issue of the *Journal*, and constitutes a very interesting survey of this field.

MERCER LECTURE—The tenth Mercer Lecture, on *The Coloration of Leather*, was delivered at the Royal Institution, London, on 9th April 1953, by Mr. J. R. Blockey and Mr. D. H. Tuck, of the National Leathersellers' College, the chair being taken by the then President, Mr. H. H. Bowen. This very useful review was subsequently published in the August issue of the *Journal*.

The eleventh Mercer Lecture is to be given at the Midland Hotel, Manchester 2, on Thursday, 8th April 1954, when Mr. N. W. Yelland will speak on *Physics in the Dyeing, Printing, and Finishing Industries*.

ANNUAL DINNER—The sixty-ninth Annual Dinner was held at the May Fair Hotel, London W.1, on 10th April 1953, the attendance being just short of 300. The principal guest was the Right Honourable the Earl of Halsbury, Managing Director of the National Research Development Corporation, who gave a very amusing and witty address ranging from the difficulties of an amateur trying to apply a dye to a fabric to those of a colour-blind man who found himself chief colour matcher in a dye works.

FASTNESS TESTS CO-ORDINATING COMMITTEE—The Committee has held five meetings during the year and has maintained its activities in the international field. The Technical Subcommittees have continued their valuable work and have made gratifying progress.

Following the meeting of I.S.O. in New York in November 1951, the A.A.T.C.C. undertook the lengthy task of setting out the agreed tests in the accepted form, and this draft was circulated to all participating members for their comments. A revised draft was then prepared by the A.A.T.C.C. and this, subject only to minor changes of spelling, was published in full in the November issue of the *Journal*. The British Standards Institution also issued the tests simultaneously in a revised form as a draft British Standard.

The I.S.O. tests incorporate the new method of assessing the results obtained in fastness testing, that is by the use of grey scales of fixed contrasts. During the year the Society has issued its Grey Scale for Assessing Staining, this being complementary to the Grey Scale for Assessing the Effect on the Pattern which had been announced previously.

During the year the B.S.I. issued the revised British Standard 1006 on "Fastness to Daylight of Coloured Textiles", this being essentially the work of the Light Fastness Subcommittee. It may be noted that this test and the draft I.S.O. test are almost identical. Consideration is now being given to the establishment of a range of pale shades which could serve as standards when testing dyes in small concentrations.

During the year the Committee has sponsored three publications in the *Journal*, two of these

dealing with the development of the grey scales, and the third being the I.S.O. tests previously mentioned.

Close contact has been maintained with overseas fastness committees. By invitation of the E.C.E., the Chairman of the F.T.C.C. attended a meeting of their working committee in Paris in March, and the Chairman and the Honorary Secretary of the F.T.C.C. were present at their third conference, which was held at Bad Kissingen, Germany, in April.

The Committee and the Subcommittees are now preparing for the next meeting of I.S.O., which will take place at Scarborough on 4–7th June 1954, immediately prior to the joint conference of the Textile Institute and the Society. At this meeting it is hoped that the draft I.S.O. tests will be confirmed and that their range will be extended by the inclusion of several tests not hitherto discussed.

DYEING AFFINITY OF REGENERATED CELLULOSE COMMITTEE— A tentative method of testing has been agreed, but no further work can be done until samples of specially modified regenerated cellulose are available.

PRESERVATION OF HISTORICAL RECORDS— The work of this Committee has continued during the year, and a number of historical records have been saved from destruction.

TERMS AND DEFINITIONS COMMITTEE— There have been five meetings of the Committee, at which over fifty definitions have been discussed. Three of the meetings have also been attended by representatives of the Textile Institute, and considerable progress has been made in collaboration with the Institute. Also, liaison has been established with the Terms and Definitions Committee of the A.A.T.C.C.

REPRESENTATIVES ON EXTERNAL BODIES— The thanks of Council are extended to members who have represented the Society on the external bodies listed on p. 231 of the July 1953 issue of the *Journal*, and Council wishes to thank them for their annual reports on the activities of these bodies.

Early in the year Mr. R. Conchar of Messrs. John Conchar & Co. felt compelled, owing to pressure of his other work, to relinquish his position as Honorary Solicitor to the Society. Council accepted Mr. Conchar's resignation with great regret, and at a small dinner party held in September, attended by the President and other officers of the Society, a silver tray was presented to Mr. Conchar in appreciation of his very valuable services to the Society during the past twenty years.

Council wishes also to thank Mr. L. E. Jones for acting as Honorary Patent Agent, and for the interest he takes in the Society's affairs.

Council records its appreciation of the various donations which have been received during the year.

This report cannot give a complete account of the Society's activities during the past year, and a more complete picture may be obtained from a perusal of the *Journal*.

The gratitude of Council must be expressed to all members who have served the Society in an honorary capacity, and in thanking them as individuals Council would also thank the firms and institutions which make it possible for them to serve the Society as they do.

Council expresses its extreme gratitude to Dr. Hooper for his excellent work during a particularly difficult period. In addition to his normal heavy duties he has had to undertake those of the Assistant Secretary. This has involved extra work, which Dr. Hooper has undertaken most readily and capably.

Finally, Council desires to express appreciation to members of the staff for their excellent work during the past year.

The Honorary Secretary (Mr. J. G. HOPKINSON), in moving the adoption of the Annual Report, which had been in members' hands during the past four weeks, paid a warm tribute to the work of the permanent staff. He referred to the heavy work which had been undertaken by Dr. C. J. W. Hooper, and to the decision to relieve him of part of this burden. Mr. Hopkinson welcomed the new Secretary, Mr. J. W. Nicholls, to the Society.

The recent formation, and affiliation to the Society, of the Dyers and Colourists Association of South Africa gave great pleasure to the Society, and the new body could be assured of the Society's keen interest in its future success and wellbeing.

The Perkin Centenary celebrations in 1956 would be on a grand scale, and the Society deeply appreciated the support of the Royal Society and the three chartered chemical organisations. Dr. C. J. T. Cronshaw had very kindly consented to become Chairman of the Perkin Centenary Organising Committee.

The golf competition, inaugurated in 1953, would become a separate function, and this year it would be held at Buxton on May 30th.

It was with great regret that he had to announce that the prospective donor of the J. B. Wilkinson Scholarship had died since the announcement of the award, and it was not quite clear what would transpire ultimately, although there were hopes that the bequest would stand.

There had been lively activity going on in all sections and they would be making a drive for increased membership.

Mr. Hopkinson spoke of the great responsibility which had been handed down to him by his predecessors, and his desire to acquit himself well in the office which he had undertaken.

Mr. J. BARRITT seconded the motion, which was carried unanimously.

ANNUAL ACCOUNTS

The Honorary Treasurer (Mr. H. JENNISON), in presenting the accounts for the year ended 31st December 1953, copies of which had been sent to members four weeks earlier, said that consideration of the Balance Sheet showed a surplus for the year of £3,336 12s. 6d. Attention should also be drawn to the £12,745 resulting from the Prepayments Scheme for the Second Edition of the *Colour Index*, which might be regarded as a very satisfactory

Balance Sheet as at 31st December 1953

31.12.52		SURPLUS AND LIABILITIES	£ s. d.	£ s. d.
		SURPLUS AND DEVELOPMENT		
26,622		Balance of Accumulated Funds at 1.1.53 ...	30,334 8 9	
3,712	30,334	*Add:* Surplus for the year to 31.12.53	3,336 12 6	33,671 1 3
1,851		FOUNDATION FUND		1,850 13 0
100		KNECHT MEMORIAL FUND		100 0 0
2,000		GEORGE DOUGLAS LECTURE FUND ...		2,000 0 0
		PREPAYMENTS SCHEME		
		Colour Index (2nd Edition)		12,745 13 10
		AMOUNTS RECEIVED IN ADVANCE		
1,845		Subscriptions		162 7 0
		CURRENT LIABILITIES		
6,436		Sundry Creditors and Amounts Accrued (per schedule)		12,116 11 0
		COLOUR INDEX (2nd EDITION)		
		NOTE— At the date of the Balance Sheet there exists a liability in respect of a proportion of expenditure incurred by the American Association of Textile Chemists and Colorists of which one-half is payable by the Society, which is not ascertainable at the date of this Balance Sheet.		
		H. JENNISON - *Honorary Treasurer*		
		J. G. HOPKINSON - *Honorary Secretary*		
£42,566				£62,646 6 1

31.12.52		ASSETS	£ s. d.	£ s. d.
		DEVELOPMENT ACCOUNT		
	20,045	INVESTMENTS AT COST ... (Market Value £21,491 3 11)		23,165 10 1
		FOUNDATION FUND		
	1,851	INVESTMENTS AT COST ... (Market Value £1,843 12 9)		1,850 13 0
		KNECHT MEMORIAL FUND		
	100	INVESTMENTS AT COST ... (Market Value £84 9 11)		100 0 0
		GEORGE DOUGLAS LECTURE		
	2,000	FUND INVESTMENTS AT COST (Market Value £1,670 3 4)		2,000 0 0
		CURRENT ASSETS		
91		Cash in Hand	30 13 1	
299		Cash in Bank	1,423 0 11	
		Cash in Bank—Colour Index (2nd Edition)	1,230 17 0	
		Colour Index (2nd Edition) Deposit Account ...	11,038 11 6	
3,630		Debtors and Prepayments (per schedule) ...	4,461 12 10	
		Stocks (excluding Colour Index 2nd Edition) (per		
2,981	6,932	schedule)	1,916 5 4	20,101 0 8
		COLOUR INDEX (2nd EDITION) DEVELOPMENT ACCOUNT		
6,395		Expenditure to date (per Accumulations Account)	7,285 10 0	
4,668		Stock of Paper	4,668 10 0	
	11,063	Work in Progress ...	2,975 0 0	14,929 0 0
		FIXED ASSETS		
		FURNITURE AND EQUIPMENT		
62		Library	56 0 0	
366		General	354 0 0	
97	525	New Colour Index... ...	90 2 4	500 2 4
	£42,566			£62,646 6 1

AUDITORS' REPORT

We have examined the above Balance Sheet with the Books and Documents of the Society produced to us. In our opinion the above Balance Sheet is properly drawn up so as to exhibit a true and fair view of the state of the Society's affairs according to the best of our information and the explanations given to us and as shown by the Books of the Society.

Bradford
1st March 1954

RAWLINSON, GREAVES & MITCHELL
Chartered Accountants
Auditors

COLOUR INDEX (SECOND EDITION) ACCOUNT

31.12.52			£ s. d.	£ s. d.
		STOCK OF PAPER 1.1.53		4,668 10 0
		TOTAL EXPENSES		
243		Wages and National Health Insurance	403 1 1	
		Printing and Publishing ...	2,975 0 0	
236		Printing, Stationery, Advertising and Postages...	386 14 10	
32		Telephone	36 8 6	
9		Electricity and Heating ...	10 15 0	
6		Sundries	17 5 1	
50		Rent, Rates and Insurance ...	67 2 0	
		Depreciation		
9		Legal Charges		
21		Audit and Accountancy ...	29 10 0	
		Bank Charges	2 0 6	
1,785		Customs Duty on Paper from America		
4		Insurance on Paper	50 15 6	
754		Freight and Carriage charges on Paper from Port to Warehouse		
4,669	7,322	Cost of Paper and Shipping charges etc. to U.K.— half cost charged to S.D.C. ...		3,978 12 6
	£7,322			£8,647 2 6

31.12.52		£ s. d.
4,669	STOCK OF PAPER 31.12.53	4,668 10 0
	WORK IN PROGRESS 31.12.53	2,975 0 0
	INVESTMENT INCOME	
	Midland Bank Ltd. Deposit Account ...	113 11 6
	BALANCE carried to Colour Index	
2,653	Accumulations Account	890 1 0
£7,322		£8,647 2 6

COLOUR INDEX (SECOND EDITION) ACCUMULATIONS ACCOUNT

		£ s. d.
4,269	ACCUMULATED TOTAL AT 31.12.52	6,395 9 0½
2,654	BALANCE brought forward, being excess of Expenditure over Income for the year ...	890 1 0
£6,923		£7,285 10 0½

		£ s. d.
	TRANSFER TO GENERAL CONSOLIDATED REVENUE ACCOUNT	
528	Expenses incurred in previous years, but not allowable per agreement with A.A.T.C.C....	
6,395	ACCUMULATED TOTAL AT 31.12.53	7,285 10 0½
£6,923		£7,285 10 0½

GEORGE DOUGLAS ACCUMULATION FUND ACCOUNT

		£ s. d.
52	LECTURE EXPENSES	57 2 2½
2	BANK CHARGES	2 0 0
151	BALANCE per Accumulations and Appropriation Account 31.12.53	151 4 4½
£205		£210 6 7

		£ s. d.
146	BALANCE per Accumulations and Appropriations Account 31.12.52	150 16 5
59	INVESTMENT INCOME 3% British Transport 1978/88	59 10 2
£205		£210 6 7

CONSOLIDATED GENERAL REVENUE ACCOUNT FOR THE YEAR ENDING 31ST DECEMBER 1953

31.12.52		EXPENDITURE	£ s. d.	£ s. d.
		GENERAL OFFICE		
514		Printing and Stationery ...	322 0 2	
189		Postages	141 10 5	
1,759		Salaries, Wages and Superannuation and Staff Expenses etc.	1,674 2 6	
278		Rent, Rates and Insurance	296 8 11	
36		Electricity and Heating ...	42 2 11	
53		Telephone	65 3 10	
21		Bank Charges	21 7 0	
63		Audit and Accountancy ...	167 0 0	
69		Travelling Expenses ...	83 15 11	
139		Sundry Expenses—Miscellaneous Committee Expenses	163 9 8	
228		Removal to and decoration of new offices ...		
66		Sundries	479 8 7½	
7	3,442	Investment Charges (Midland Bank Executor and Trustee Co. Ltd.) ...	3 17 6	3,461 7 5½
	775	SECTIONAL EXPENSES ...		773 6 4
	48	DEPRECIATION ON GENERAL OFFICE FURNITURE ...		45 8 11
40		MERCER LECTURE EXPENSES	57 6 1	
20	20	*Less*— Donation	20 0 0	37 6 1
		JOURNAL		
500		Stock— 1st January 1953	580 0 0	
7,548		Printing and Publishing ...	8,547 1 7	
523		Postages	581 18 10	
1,097		Salaries, Honoraria and Wages	1,193 14 9	
361		Own Journals	156 10 0	
323		Abstractor's Fees and Book Reviews	295 17 1	
82		Miscellaneous Publications	16 19 9½	
18		Rent, Rates and Insurance	82 0 0	
30		Electricity and Heating ...	18 0 0	
71	10,553	Telephone	30 0 0	
		Printing and Stationery ...	104 9 0½	11,606 11 1
66		ANNUAL MEETING AND DINNER		
35	101	Meeting	60 1 8½	
		Dinner	50 18 11½	111 0 8
	37	DONATIONS AND SUBSCRIPTIONS		32 17 6
	49	REPAIRS AND RENEWALS...		21 4 6½
		BAD DEBTS		89 3 6
		LIBRARY UPKEEP		34 3 8
	114	BYE-LAWS		
	613	COLOUR INDEX 2nd Edition Expenses incurred but not allowable per agreement with A.A.T.C.C.		45 13 2
		FASTNESS TESTS ACCOUNT		
1,491		Stock— 1st January 1953	1,510 7 4	
585		Materials purchased ...	274 7 9	
8		Postages	13 15 9	
114		Loose Patterns and Bunches	37 1 6	
21		Fastness Tests Report ...		
61		International Conference		
	2,280	Expenses		1,835 12 4
		IDENTIFICATION OF DYES ACCOUNT		
38		Stock— 1st January 1953	4 6 0	
		Printing	114 18 3	
3	41	Sundry Expenses	2 9 0	121 13 3
		WOOL DYEING ACCOUNT		
14		Stock— 1st January 1953	500 17 7	
		Printing and Publishing ...		
6		Postages	5 11 8½	
23	643	Royalties	17 3 6	523 12 9½
		REPLIKA COLOUR INDEX AND SUPPLEMENT		
		Stock— 1st January 1953		
308		Index and Supplement ...	72 10 11	
		Printing and Binding ...		
44	352	Sundry Expenses	6 8 4	78 19 3
	3,712	SURPLUS FOR THE TWELVE MONTHS		3,336 12 6
	£22,780			£22,154 13 0

31.12.52		INCOME	£ s. d.	£ s. d.
		SUBSCRIPTIONS		
6,743		Members	6,666 6 11	
81	6,824	Juniors	83 15 3	6,750 2 2
		JOURNAL ACCOUNT		
4,514		Sale of Journals and Reprints	4,735 3 8	
102		Combined Reports etc. ...	35 19 8	
6,642		Advertisements	6,897 3 11	
580	11,838	Stock— 31st December 1953	550 0 0	12,218 7 3
		INVESTMENT INCOME (GROSS)		
33		Foundation Fund	32 18 6	
639	672	Development Fund ...	597 1 10	630 0 4
	152	DONATIONS		152 2 0
		FASTNESS TESTS ACCOUNT		
11		Grey Scales	204 2 8	
		Staining Scales	154 5 6	
17		Reports	27 15 2	
393		Material	170 19 2	
362		Bunches and Loose Patterns	496 15 0	
1,510	2,293	Stock— 31st December 1953	514 17 4	1,568 14 10
		IDENTIFICATION OF DYES ACCOUNT		
75		Sales	54 5 2	
13		Royalties		
4	92	Stock— 31st December 1953	112 10 0	166 15 2
		WOOL DYEING ACCOUNT		
171		Sales	135 1 8	
		Advertising		
501	672	Stock— 31st December 1953	414 0 0	549 1 8
		REPLIKA COLOUR INDEX AND SUPPLEMENT		
464		Sales— Index and Supplement	68 13 7	
73	537	Stock— 31st December 1953	50 16 0	119 9 7
	£22,780			£22,154 13 0

SCHEDULE OF DEVELOPMENT ACCOUNT INVESTMENTS

	Balance 1952	Alterations	Balance 1953	Market Values 1952	Market Values 1953	
	£ s. d.		£ s. d.	£ s. d.	£ s. d.	
£2,763 3s. 8d. 3% British Transport Guaranteed Stock 1978/88	2,598 17 0		2,598 17 0	2,126 14 10	2,326 5 1	84½
£750 2½% Funding Stock 1956/61	750 0 0		750 0 0	698 8 9	734 1 3	97½
£546 3½% Conversion Loan	557 0 0		557 0 0	428 5 4	465 9 5	85½
£1,255 2½% Defence Bonds	1,255 0 0		1,255 0 0	1,255 0 0	1,255 0 0	Par
£2,749 16s. 3% Savings Bonds 1960/70	2,750 0 0		2,750 0 0	2,351 1 7	2,509 3 10	91½
£750 2½% Exchequer Bonds 1955	750 0 0		750 0 0	739 4 4	749 10 3	99⅞
500 National Savings Certificates 15s. Units 7th Issue	375 0 0		375 0 0	375 0 0	375 0 0	—
£2,000 3% British Gas Stock 1990/95	2,000 0 0		2,000 0 0	1,528 15 0	1,705 0 0	85½
£2,017 11s. 6d. 3% Treasury Stock 1966 or after	1,957 1 0		1,957 1 0	1,369 8 7	1,505 12 3	74½
£901 3s. 6d. 3% Southern Rhodesian 1971/73	910 13 9		910 13 9	720 18 10	770 10 1	85½
York County Savings Bank	3,133 10 8	£79 13s. Interest	3,213 3 8	3,133 10 8	3,213 3 8	
£1,500 12s. 9d. 3% British Electricity Guaranteed Stock 1968/73	1,495 19 0		1,495 19 0	1,259 11 11	1,338 7 8	89½
Post Office Savings Bank	517 16 8	£12 18s. 6d. Interest	530 15 2	517 16 8	530 15 2	—
£1,000 Chesterfield Corporation 3% Stock 1960	993 15 0		993 15 0	930 0 0	985 0 0	98½
Midland Bank Ltd. Deposit Account	— — —	£3,000 invested during year. £28 5s. 3d. Interest.	3,028 5 3		3,028 5 3	—
	£20,044 13 1		£23,165 9 10	£17,427 16 6	£21,491 3 11	

SCHEDULE OF DEBTORS AND PREPAYMENTS

	31.12.52	31.12.53
	£ s. d.	£ s. d.
Advertisements	1,828 8 10	2,145 15 8
Combined Reports	1 7 6	2 15 3
Journal	881 4 9	1,510 2 0
Miscellaneous Publications	2 6	8 0 6
Wool Dyeing	42 17 6	53 10 0
Tinctorial Arts Conference	9 12 6	7 14 9
Reprints	91 6 7	23 3 5
Fastness Tests, etc.—		
Loose Patterns	67 10 0	
Fastness Tests	67 0 8	63 17 7
Grey Scale	1 12 0	127 11 5
Replika Colour Index—Supplement	189 3 7	72 18 11
Review of Textile Progress	2 2 6	
Abstracts	10 17 6	2 10 7
Sundries	9 6	
Freight, Insurance and Packing	27 8 6	21 10 0
Photochemistry Symposium	25 6 8	9 2 6
Identification of Dyes	16 5 2	25 16 3
Membership	22 15 3	34 1 6
Postage	8 0	
Rates and Insurances in advance	28 8 9	98 16 3
Income Tax Repayment Due	101 12 8	96 5 8
Subscriptions and Donations	150 0 0	150 0 0
Colour Index (2nd Edition)—General	46 10 6	
Printing and Stationery		14 7
Repairs and Renewals	7 10 0	7 10 0
	£3,620 1 5	£4,461 12 10

SCHEDULE OF CREDITORS AND AMOUNTS ACCRUED

	31.12.52	31.12.53
	£ s. d.	£ s. d.
Printing—Stationery		51 16 0
Accountancy—Professional Charges	63 0 0	170 0 0
General Expenses	39 13 5	20 19 4
Sectional Expenses	26 9 11	30 14 8
Journal Expenses—		
Printing and Publishing	1,323 6 4	3,782 11 7
Postages	75 19 9	173 9 10
Reprints	26 5 0	
Amounts received in advance	78 18 6	76 19 6
Miscellaneous Committees	15 0 11	15 17 11
Royalties *Wool Dyeing*		17 3 6
Fastness Tests	100 0 0	
Colour Index (2nd Edition)		
General	21 12 0	
A.A.T.C.C.	4,668 10 0	4,668 10 0
Printing *Symposium*		133 8 8
N.C.I. Printing and Publishing		2,975 0 0
	£6,436 15 10	£12,116 11 0

SUMMARY OF STOCK

	31.12.52	31.12.53
	£ s. d.	£ s. d.
Fastness Tests and Materials	1,510 7 4	514 17 4
Journals	" 0 0	550 0 0
Photochemistry Symposium	15 17 4	12 16 0
Replika *Colour Index* and Supplement	72 10 11	50 16 0
Identification of Dyes	4 6 0	112 10 0
Wool Dyeing	309 17 7	414 0 0
Stationery	181 5 9	100 0 0
Combined Reports, etc.	143 17 1	153 5 0
Tinctorial Arts Conference	11 4 0	8 1 0
	£2,980 6 0	£1,916 5 4
COLOUR INDEX (2nd Edition)		
Stock of Paper	4,668 10 0	4,668 10 0
Work in Progress	— — —	2,975 0 0
	£4,668 10 0	£7,643 10 0

SCHEDULE OF FOUNDATION FUND INVESTMENTS

	Balance 1952	Balance 1953	Market Values 1952	Market Values 1953	
	£ s. d.	£ s. d.	£ s. d.	£ s. d.	
£1,245 2½% Defence Bonds	1,245 0 0	1,245 0 0	1,245 0 0	1,245 0 0	—
1,000 National Savings Certificates 10s. units 8th issue	500 0 0	500 0 0	500 0 0	500 0 0	—
£68 8s. 6d. 3% Savings Bonds 1960/70	69 9 0	69 9 0	55 10 0	62 8 9	91½
Midland Bank Ltd. (Cash awaiting investment) No. 2 Account	36 4 0	36 4 0	36 4 0	36 4 0	—
	£1,850 13 0	£1,850 13 0	£1,839 14 0	£1,843 12 9	

GEORGE DOUGLAS LECTURE FUND

£1,983 17s. 0d. 3% British Transport Stock 1978/88	£2,000	£2,000	£1,522 13 1	£1,670 3 4	84½

KNECHT MEMORIAL FUND

£98 16s. 6d. 3% Southern Rhodesia 1971/73	£100 0 0	£100 0 0	£79 1 2	£84 9 11	85½

SCHEDULE OF OFFICE FURNITURE AND FITTINGS

	General		Colour Index (2nd Edition)		Library	
	1952	1953	1952	1953	1952	1953
	£ s. d.	£ s. d.	£ s. d.	£ s. d.	£ s. d.	£ s. d.
Balance 1.1.52	378 9 8	366 11 5	96 0 0	97 2 4	65 2 6	62 2 6
Additions	29 1 9	26 15 0	12 2 4	4 0 0	4 0 0	
	407 11 5	393 6 5	108 2 4	101 2 4	69 2 6	62 2 6
Less Depreciation @ 10%	41 0 0	39 6 5	11 0 0	11 0 0	7 0 0	6 2 6
TOTAL PER BALANCE SHEET	£366 11 5	£354 0 0	£97 2 4	£90 2 4	£62 2 6	£56 0 0

send-off for the scheme. The major portion of this had been placed on deposit with the Midland Bank until called for. The increase in current liabilities was due largely to expenditure on the new *Colour Index* inclusive of paper amounting to £7,643, details of which were given in the Schedule of Creditors. The total amount expended on the new *Colour Index* to date, including these items, as shown in the Balance Sheet, was £14,929.

As regards the Consolidated General Revenue Account, there was little difference between this year and last. Income from membership subscriptions at the year end was slightly down, owing to the failure of some members to pay as promptly as they might, but this had now been rectified. Once again we were greatly indebted for donations amounting to £152.

The Schedule of Development Account Investments showed a definite increase in market value compared with 1952. It was the accepted custom in the majority of Balance Sheets to enter investments at cost in order to avoid instability, but it was interesting to note that, were the realisable value of these investments taken into account, the actual surplus for the year would be comparatively small.

Mr. J. V. SUMMERSGILL seconded the motion, and on a show of hands it was declared carried.

ELECTION OF PRESIDENT

Mr. FRED SMITH, in proposing that Mr. F. L. Goodall be President for a second year, paid high tribute to Mr. Goodall's good work for the Society.

Mr. A. S. FERN seconded the motion, which was carried with acclamation.

Mr. F. L. GOODALL, responding, spoke of the work being very near to his heart, and in difficult times he had been supported by an upsurge of enthusiasm and good feeling.

ELECTION OF PRESIDENT-ELECT

Mr. F. SCHOLEFIELD, in proposing Mr. FRED SMITH to be President-elect, spoke of the confidence and goodwill of his fellow-members. Mr. A. W. CARPENTER seconded. The President paid tribute to Mr. Smith and on the motion being put to the vote this was carried with acclamation.

Responding, Mr. FRED SMITH spoke of this climax of joy to happy years of work for the Society.

ELECTION OF A VICE-PRESIDENT

The President declared that, no other nomination having been received, Mr. H. A. Turner was elected a Vice-president. He congratulated him and welcomed him to this office.

ELECTION OF ORDINARY MEMBERS OF COUNCIL

The President declared Messrs. A. S. Fern, H. Hampson, J. W. Reidy, and A. G. Tyler, the only nominations received, to be elected Ordinary Members of Council.

VOTE OF THANKS TO RETIRING VICE-PRESIDENTS AND ORDINARY MEMBERS OF COUNCIL

Dr. C. M. WHITTAKER spoke of the untiring energy devoted in service to the Society and moved a vote of thanks to the four retiring Vice-presidents (Mr. W. A. Edwards, Mr. R. J. Hannay, Dr. H. A. Thomas, and Mr. G. M. Williams) and the four retiring Ordinary Members of Council (Mr. D. Hanson, Mr. H. C. Olpin, Dr. C. B. Stevens, and Mr. H. A. Turner).

Mr. A. P. KERSHAW seconded, and the motion was carried with enthusiasm.

ELECTION OF HONORARY TREASURER

The President declared that, no other nomination having been received, Mr. H. Jennison was re-elected Honorary Treasurer.

ELECTION OF HONORARY SECRETARY

The President declared that, no other nomination having been received, Mr. J. G. Hopkinson was re-elected Honorary Secretary.

APPOINTMENT OF AUDITORS

Mr. C. O. CLARK proposed, Mr. H. TURNER seconded, and it was agreed that Messrs. Rawlinson, Greaves & Mitchell, Chartered Accountants, be re-appointed Auditors.

ELECTION OF VICE-PRESIDENTS FOR 1955

The President moved the adoption of the following scheme to facilitate the change in the number of Vice-presidents at the ballot in 1955—

> The scheme shall become operative at the 1955 Annual General Meeting to facilitate the functioning of the new Bye-law that there be six Vice-presidents to hold office for six years, with one retiring annually.
>
> In 1955 there will be four retiring Vice-presidents, and of the resulting vacancies, one shall constitute the annual vacancy for 1955, and the remaining three shall be regarded as casual with terms of office of three, two, and one year respectively, determined in decreasing order of number of votes cast at the 1955 ballot.

This was carried.

VOTE OF THANKS TO PRESIDENT

Mr. R. J. HANNAY spoke of the arduous work of the President and moved a hearty vote of thanks to him for presiding over the meeting. Mr. E. ROLFE seconded, and the vote of thanks was carried with acclamation.

Annual Dinner

The Seventieth Annual Dinner of the Society was held at the Midland Hotel, Manchester 2, on Friday evening, 30th April 1954, under the presidency of Mr. F. L. Goodall.

The principal guest was was Mr. P. F. Crosland. Among other guests were Dr. R. V. Bowden (*Principal of the Manchester College of Technology*), Mr. C. R. Egerton (*Prime Warden of the Worshipful Company of Dyers*), Mr. W. Crossley (*Chairman of the Confederation of Textile Dyers and Finishers*), Mr. J. Brear (*Chairman of the Victoria Section of the Society of Dyers and Colourists of Australia*), Mr. H. Gosling (*President of the Oil and Colour Chemists Association*), Mr. J. Davidson Pratt (*Director and Secretary of the Association of British Chemical Manufacturers*), Mr. W. R. Mathers

(*Chairman of the Dyers and Finishers Association*), Mr. C. G. Hulse (*Secretary of the Dyestuffs Control, Board of Trade*), Mr. J. Wilson (*Director of Research of the British Rayon Research Association*), Mr. J. Barritt (*Honorary Member*), Mr. H. A. Turner (*Gold Medallist*), Mr. L. E. Jones (*Honorary Patent Agent*), Mr. P. Caldwell, Mr. A. D. Ferns, Mr. C. F. Gysin, Mr. F. A. Helme, Mr. C. Paine, Mr. C. A. Staehelin, Dr. W. Stauffacher, Mr. T. M. Willcox, Dr. C. J. T. Cronshaw and Mr. F. Scholefield (*Past Presidents*), Mr. Fred Smith (*President-elect*), and Mr. G. S. J. White (*Chairman of the Manchester Section*).

The following officers and other leading members of the Society also were present—Mr. H. H. Bowen (*Immediate Past President*), Dr. C. M. Whittaker (*Past President*); Mr. G. B. Angus, Mr. C. O. Clark, and Mr. H. Foster (*Vice-presidents*); Dr. G. T. Douglas, Mr. A. S. Fern, Mr. N. Hamer, Mr. H. Hampson, Mr. A. P. Kershaw, Mr. J. W. Reidy, and Mr. J. V. Summersgill (*Ordinary Members of Council*); Mr. F. Atack, Mr. A. W. Carpenter, Mr. H. R. Hadfield, Mr. S. R. Meadows, Mr. R. A. Peel, Mr. J. Porter, Mr. H. W. Taylor, and Mr. H. Turner (*ex-officio Members of Council*); Mr. H. Jennison (*Honorary Treasurer*); and Mr. J. G. Hopkinson (*Honorary Secretary*).

The total number present was 409, a record in the history of the Society.

"THE SOCIETY OF DYERS AND COLOURISTS"

After the toast of "Her Majesty the Queen", that of "The Society of Dyers and Colourists" was proposed by—

Mr. P. F. CROSLAND, who referred to his first connection with the Society, when, as a student in the Department of Dyeing and Colour Chemistry at Leeds University, he had expanded a sparse income in making abstracts of patents and original papers for the Society's *Journal*. Unfortunately, the Editor had a heavy blue pencil and a strong wrist, and the financial return was sadly out of correspondence with the time taken.

Later on, as assistant to Professor A. G. Green, F.R.S., then head of the department, he was present at a private lunch in the Great Northern Hotel at Bradford, when Green first suggested that an up-to-date *Colour Index* was urgently needed, and that the institution which would accept and fulfil that high responsibility would gain the thanks of the dyestuff industry and bring honour to its own reputation. As everybody making and using dyes knows, the work was later taken in hand by a fellow student, F. M. Rowe, who when he came to occupy the Chair at Leeds fully maintained the high reputation which it gained during Green's tenure. The First Edition of the *Colour Index* abundantly justified the most sanguine hopes of its sponsors, and the Second Edition, now so badly needed, will certainly add lustre to the high reputation of the Society of Dyers and Colourists wherever, throughout the world, dyes are made or used.

"OUR GUESTS"

Mr. F. L. GOODALL (*President of the Society*) responded to the toast and also proposed the toast of "Our Guests". He first thanked the Chief Guest, Mr. P. F. Crosland, a Leeds graduate of the vintage of Baddiley, Scholefield, and Whittaker, a leading industrialist, and above all a practical colourist.

The Society was especially glad to welcome Mr. C. R. Egerton, Prime Warden of the Worshipful Company of Dyers, which had shown a sustained interest in the affairs of the Society, notably by the presentation of the Presidential Badge. Council had recently decided to present small badges to retiring presidents, and they had already been sent to the seven surviving past presidents, one of whom (Mr. H. H. Bowen) the Society would like to congratulate on his recent engagement to be married. The President felt that this was a suitable occasion to announce that Mr. Clifford Paine had accepted Council's nomination for election as President-elect in 1955, as Mr. Fred Smith would be able to serve as President for only one year.

Two visitors who had travelled far to attend the Dinner were Mr. J. Brear and Mr. A. Murray of the Society of Dyers and Colourists of Australia, the former being Chairman of the Victoria Section. We took great pleasure in their presence with us, and were proud of our close ties with the affiliated society in Australia. We were gratified by the recent affiliation to our Society of the newly formed Dyers and Colourists Association of South Africa, and we hoped that there would be mutual co-operation between the two affiliated bodies themselves.

Two other overseas visitors were Mr. C. A. Staehelin and Dr. W. Stauffacher from Basle, Switzerland, both of whom had travelled the world in the interests of the dyemaking industry.

It was a particularly welcome privilege to be able to entertain the President of the Oil and Colour Chemists Association (Mr. H. Gosling). The work of O.C.C.A. was in many respects complementary to our own, and we had a common interest in colour, so that we earnestly hoped that we might look forward to mutually beneficial co-operation in the future.

An always welcome guest was Mr. Charles G. Hulse, of the Board of Trade, who was responsible for the smooth working of the Dyestuffs (Import Regulation) Act, 1920. We were pleased to welcome also Mr. J. Davidson Pratt, Director and Secretary of the Association of British Chemical Manufacturers, and Mr. J. Wilson, Director of Research of the British Rayon Research Association.

The users of dyes were very well represented by Mr. P. Caldwell, Mr. W. Crossley (Chairman of the Confederation of Textile Dyers and Finishers and of the Flat Dyed Rayon Association), and Mr. W. R. Mathers (Chairman of the Dyers and Finishers Association).

The Society was always grateful for the support and active help of the dyemaking firms, who were

JOHN BARRITT
B.Sc. A.R.C.S. A.R.I.C. F.S.D.C.
Honorary Membership in recognition of outstanding services to the Society, most notably as Honorary Secretary from 1946 to 1953

HAROLD AUGUSTUS TURNER
M.Sc. F.R.I.C. F.T.I. F.S.D.C.
Gold Medal for valuable researches contributed to the tinctorial industries

JOHN GARNETT HOPKINSON
M.A. A.R.I.C.
Honorary Secretary of the Society

JOHN WARNER NICHOLLS
F.C.I.S.
Secretary of the Society

ALFRED WILLIAM CARPENTER
Silver Medal for valuable services to the Society

MARJORIE FORBES
Bronze Medal for valuable services to the Society

HARRY ROSE HADFIELD
M.Sc.Tech.
Bronze Medal for valuable services to the Society

represented tonight by Mr. A. D. Ferns, Mr. C. F. Gysin, Mr. F. A. Helme, and Mr. T. M. Willcox.

Finally, but very far from least, we extended a very warm welcome to Dr. B. V. Bowden, Principal of the Manchester College of Technology. Dr. Bowden was a physicist of repute who had worked on radioactivity with Lord Rutherford, on the development of radar, and on digital computers or "electronic brains", on which he had written a book—*Faster than Thought*.

Dr. B. V. Bowden (*Principal of the Manchester College of Technology*) in response to the toast referred to the development of the dyeing department at the College over more than fifty years and to its three heads—Mr. J. Hübner, Dr. Edmund Knecht, and Mr. Fred Scholefield—who had taken such an active interest in the affairs of the Society. The new dyehouse and other extensions, begun in 1950 and now nearing completion, would make the department one of the best equipped in the world. Dr. Bowden appealed to members of the Society and to the trade for active assistance and support in making the College a leading centre of training and research in the science and technology of dyes and their application.

PRESENTATION OF DIPLOMA OF HONORARY MEMBERSHIP TO MR. J. BARRITT

Dr. C. J. T. Cronshaw, introducing Mr. Barritt, said that the award of Honorary Membership was a cherished honour, which was extremely well merited in the case of John Barritt, B.Sc., A.R.C.S., A.R.I.C., F.S.D.C. The Society had for long been able to call on the services of willing workers, but all members knew the exceptional amount of time and energy which Mr. Barritt had devoted to the welfare of the Society. After a period of service on the Publications Committee, Mr. Barritt had become Honorary Secretary of the Society in 1947. His resignation from this office in 1953 was caused by the pressure of his work as Chairman of the Editorial Panel and a Part I editor of the Second Edition of the *Colour Index* and increasing duties with the Wool Industries Research Association. Since his resignation he had become chairman of the Publications Committee, and in this way his experience would still be available to the Council. Apart from his direct work for the Society, Mr. Barritt had served the industry and the technology of dyeing for over twenty-five years.

Mr. J. Barritt said that it was difficult to find the proper words in which to thank the President and members of Council for the signal honour, which he now shared with the Prime Warden of the Worshipful Company of Dyers, Dr. Cronshaw, and Mr. H. Jennison, but he was most grateful and appreciative. He also expressed his thanks for Dr. Cronshaw's generous tribute and for the kindness he had always received from all members of the Society during his period of office as Honorary Secretary. He would like to acknowledge the generous attitude of his employers, the Wool Industries Research Association, in granting him the necessary facilities to serve the Society.

He had had the honour to serve under five presidents—Dr. C. M. Whittaker, the late George Holden, Mr. Fred Scholefield, Mr. H. H. Bowen, and Mr. F. L. Goodall—to all of whom he paid tribute for their work for the Society. He regretted that he had had only a few months under Mr. Goodall, but was glad that he had been allowed to play some part in the selection of his successor, Mr. John Hopkinson, a young man who, he felt sure, would give devoted and valuable service to the Society.

Finally, Mr. Barritt said that, as the newest Honorary Member, he intended to continue to serve the Society so far as he was able so to do, a Society which, in some indefinable way, demanded the best in us and was at the same time so very dear to us all.

PRESENTATION OF THE SOCIETY'S GOLD MEDAL TO MR. H. A. TURNER

Mr. F. Scholefield, in presenting Mr. Turner, said that, over the past twenty years, Mr. Turner had contributed twenty to thirty papers on many different aspects of textile and tinctorial technology. In 1934–1935 he had been awarded the Worshipful Company of Dyers Research Medal for a paper on *The Effect of Reduced Vat Dyes upon the Hypochlorite Oxidation of Cellulose*, and had followed with a series of papers on the behaviour of vat dyes as activators of photochemical reactions, particularly the oxidation of cellulose. More recently he had embarked upon the examination of some of the complex phenomena associated with the processes of textile printing, and had brought forward a wealth of factual data on the diffusion of colouring matters from the thickener into textile substrates, which pioneer work had already had a stimulating effect on this branch of tinctorial technology.

Mr. Turner had served the Society on Council, the Publications Committee, the Terms and Definitions Committee, and the Joint *Review of Textile Progress* Committee. He was a most active member of the Diploma Committee, and an outstanding teacher of technology in Manchester.

Mr. H. A. Turner expressed his very sincere appreciation of the honour. Apart from any question of assessing true merit, the occasion gave him undiluted personal pleasure, firstly to receive the medal at the hands of Mr. F. L. Goodall, secondly to be presented by his friend and mentor Mr. F. Scholefield, and finally to have received so many agreeable good wishes from members.

Members were no doubt expecting from him a learned discourse on one of the abstruse problems in technology occupying their minds at that very moment. There were precedents; for at the best known of all symposia it had been decided to waive the music and dancing girls, to drink only when so inclined, and to spend all night discussing one subject of supreme importance. On that occasion one of the guests had fallen asleep and had awakened at daybreak to find that Socrates had just talked the last two of the party into unconsciousness. He would not carry out the implied threat, but members would agree that Socrates was a shining example of an educator,

for he had sought to help his disciples to discover what was within themselves rather than to give them a surfeit of instruction. If he (Mr. Turner) had any special merit, it was that he had encouraged all the young men who had been his collaborators to do worthwhile work. Every one of them had made a substantial personal contribution to the whole, and he therefore regarded them all as sharing in the award, which would be as great an inspiration to them as it was to himself.

He would conclude with two wishes—that the Society would long continue its fruitful and honourable career and that the award would further encourage research into textile-printing processes. This field was a Tom Tiddler's ground—whoever explored it seriously and honestly could not fail to turn up something of value: Urquhart, Meggy, and others, like Shadrach and his companions, had passed unscathed through the steamer, and had emerged with new knowledge; Derry and Higginbotham had walked delicately along the doctor's edge with shear profit; and Turner and his co-workers had combined philately with cookery, sticking their stamps in the appointed places and steaming until tender, with the present happy event as the result.

AWARD OF OTHER MEDALS

The President referred briefly to the award of the Perkin Medal to Dr. A. Zitscher, of the Research Medal of the Worshipful Company of Dyers to Dr. R. H. Peters, of the Society's Silver Medal to Mr. A. W. Carpenter, and of the Society's Bronze Medal to Miss M. Forbes and Mr. H. R. Hadfield [see pp. 293–295], and congratulated the recipients.

Some Aspects of the Chemistry and Textile Uses of Sequestering Agents

J. V. SUMMERSGILL

Meetings of the West Riding Section held at the Victoria Hotel, Bradford, on 26th February 1953, Mr. S. L. Peel in the chair; and of the Northern Ireland Section held at the Royal Avenue Hotel, Belfast, on 4th December 1953, Mr. J. Montgomery in the chair

Certain compounds which are capable of forming complexes with metals are potentially useful for the prevention of undesirable effects due to metals frequently encountered in textile processing. The nature of the reaction between such compounds and metals is discussed with reference to derivatives of iminodiacetic acid and complex phosphates. Some applications of these compounds in the prevention of troubles caused by calcium, magnesium, iron, copper, and some other metals are outlined.

INTRODUCTION

In many treatments to which textiles are subjected, trouble often arises from traces of certain metals. These effects are caused when the metal is present in the usual form of a cation (positively charged). Faults caused by metals can often be avoided by the use of certain chemicals which react with the metal in such a way that it becomes part of a complex anion (negatively charged). When the metal is in this form, its tendency to cause undesirable results is inhibited or prevented. Such compounds are referred to as *sequestering agents*.

COMMERCIAL SEQUESTERING AGENTS

To be useful commercially as a sequestering agent a compound must meet certain requirements. The main ones are—

(i) It must form a complex which is stable under the conditions of use

(ii) It must be suitable for handling in bulk

(iii) It must be capable of being manufactured economically and in commercial quantities. Certain compounds which have excellent sequestering properties require expensive raw materials or are difficult to manufacture, which renders their price prohibitive

Any compound which converts a metal into part of a negatively charged ion is potentially useful as a sequestering agent. The compounds which have been marketed commercially chiefly for this purpose may be divided into two types—

(*1*) *The Complex Phosphates*

The two most important compounds of this type are probably sodium triphosphate (tripolyphosphate) $Na_5P_3O_{10}$ and glassy sodium metaphosphate. The latter compound is generally referred to as "sodium hexametaphosphate", which implies that the formula is $Na_6P_6O_{18}$. It is generally recognised, however, that this formula is incorrect, and that the molecular chain contains many more $NaPO_3$ units. Where the name "sodium hexametaphosphate" is used subsequently in this paper, it is done for convenience and with this reservation. Sodium triphosphate is generally manufactured by heating an intimate mixture of disodium hydrogen orthophosphate and monosodium dihydrogen orthophosphate—

$$2\,Na_2HPO_4 + NaH_2PO_4 = Na_5P_3O_{10} + 2H_2O$$

"Sodium hexametaphosphate" is prepared by allowing molten sodium metaphosphate to cool rapidly below 200°C., whereby devitrification is avoided and the metaphosphate glass is obtained.

Other phosphates which possess some metal-sequestering properties, but which are of more limited interest in this respect, are normal sodium pyrophosphate $Na_4P_2O_7$ and disodium dihydrogen pyrophosphate $Na_2H_2P_2O_7$.

(*2*) *Derivatives of Iminodiacetic Acid*

$NH(CH_2{\cdot}COOH)_2$

These compounds contain at least one $-N(CH_2{\cdot}COOH)_2$ group, and may be prepared by two general methods—

(*a*) Condensation of an amine with formaldehyde and hydrocyanic acid to give the nitrile, which is subsequently hydrolysed—

$$R{\cdot}NH_2 + 2\ HCHO + 2HCN = R{\cdot}N(CH_2{\cdot}CN)_2 + 2\ H_2O$$

$$R{\cdot}N(CH_2{\cdot}CN)_2 \xrightarrow{+4H_2O} R{\cdot}N(CH_2{\cdot}COOH)_2$$

(*b*) Condensation of an amine with an *a*-halogenated fatty acid in the presence of an acid-binding agent—

$$R{\cdot}NH_2 + 2Cl{\cdot}CH_2{\cdot}COOH = R{\cdot}N(CH_2{\cdot}COOH)_2 + 2HCl$$

It is easy to see that a large number of compounds of this type can be prepared, but the two which have found most use commercially are—

(*a*) ETHYLENEDIAMINETETRA-ACETIC ACID—
$N(CH_2{\cdot}COOH)_2{\cdot}CH_2{\cdot}CH_2{\cdot}N(CH_2{\cdot}COOH)_2$

(*b*) NITRILOTRIACETIC ACID—
$N(CH_2{\cdot}COOH)_3$

Since the free acids are sparingly soluble in water, they are usually marketed either as their sodium salts or as mechanical mixtures of the free acids and sodium carbonate.

These two acids are frequently mentioned later, and when their uses are described it is in reference to their commercial formulations.

THE MODE OF ACTION OF SEQUESTERING AGENTS

The mode of action of "sodium hexametaphosphate", ethylenediaminetetra-acetic acid, and nitrilotriacetic acid with calcium are illustrated by the following equations—

(i)

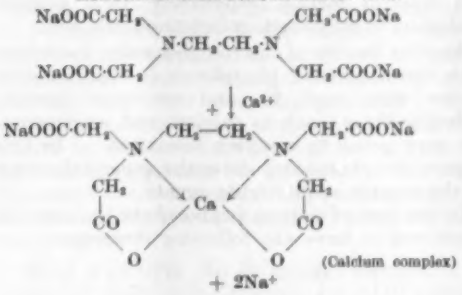
SODIUM HEXAMETAPHOSPHATE

$$Na_4P_6O_{18} \rightleftharpoons 2Na^+ + Na_4P_6O_{18}^{2-}$$

$$Na_4P_6O_{18}^{2-} + Ca^{2+} \rightarrow Na_2CaP_6O_{18}^{2-} + 2Na^+$$

(ii)

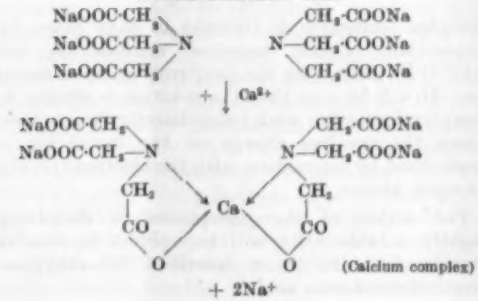
ETHYLENEDIAMINETETRA-ACETIC ACID



$$+ 2Na^+$$

(iii)

NITRILOTRIACETIC ACID



$$+ 2Na^+$$

It will be seen that in each case, if the calcium reacted completely with the sequestering agent, the concentration of calcium ion would be zero.

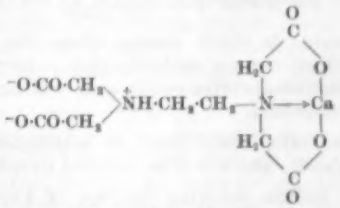
In the case of organic sequestering agents, the detailed mode of action may be discussed by reference to ethylenediaminetetra-acetic acid, which has been extensively studied in recent years. The following aspects have an important bearing on its practical applications in textile treatments. If the anion of the acid is written as Y^{4-}, the formation of the calcium complex may be represented as follows—

$$Ca^{2+} + Y^{4-} \rightleftharpoons CaY^{2-}$$

$$\frac{[CaY^{2-}]}{[Ca^{2+}]\ [Y^{4-}]} = K$$

K is referred to as the *stability constant* of the complex, and has a value of $3{\cdot}9 \times 10^{10}$ for calcium [1].

At lower pH values another complex—



is said to be formed to a limited extent—

$$Ca^{2+} + HY^{3-} \rightleftharpoons CaHY^-$$

$$\frac{[CaHY^-]}{[Ca^{2+}]\ [HY^{3-}]} = K = 3{\cdot}2 \times 10^3$$

This is less stable than the CaY^{2-} complex, as shown by the lower value for K in this case.

Ethylenediaminetetra-acetic acid forms complexes with alkaline-earth and "heavy" metals, and even with lithium and sodium weak complexes are formed. In the cases of bivalent and tervalent metals the complexes are of the types $M^{II}Y^{2-}$ and $M^{III}Y^-$ respectively, resulting from one atom of the metal and one molecule of the acid. Some values of the stability constant K are given in Table I[1,2]. The higher the value of K, the more stable is the complex.

TABLE I

Stability Constants of Metal–Ethylenediaminetetra-acetic Acid Complexes (log K)

Na^+	1·66	Co^{2+}	16·10
Li^+	2·79	Cd^{2+}	16·48
Ba^{2+}	7·76	Zn^{2+}	16·58
Sr^{2+}	8·63	Pb^{2+}	18·20
Mg^{2+}	8·69	Cu^{2+}	18·38
Ca^{2+}	10·59	Ni^{2+}	18·45
Mn^{2+}	13·47	Cr^{3+}	24·00
Fe^{2+}	14·22	Fe^{3+}	25·00

The values of the stability constant K are of importance in determining the solubilising action of ethylenediaminetetra-acetic acid on a sparingly soluble salt, e.g. calcium oxalate. If the salt dissolves completely, the reaction may be written—

$$Y^{4-} + Ca^{2+} \rightleftharpoons CaY^{2-}$$

and $$\frac{[CaY^{2-}]}{[Ca^{2+}] \times [Y^{4-}]} = K = 3{\cdot}9 \times 10^{10}$$

If the calcium oxalate is present to the extent of 1 g.-mol./litre, then at equilibrium $[CaY^{2-}] = 1$, since K is very large and the calcium ion is

converted almost completely to the complex ion CaY^{2-}. If the concentration of ethylenediaminetetra-acetic acid is such that at equilibrium $[Y^{4-}] = 1$, then—

$$[Ca^{2+}] = \frac{1}{3{\cdot}9 \times 10^{10}}$$

If the calcium oxalate is dissolved completely, then $[C_2O_4^{2-}] = 1$, and—

$$[Ca^{2+}] \times [C_2O_4^{2-}] = \frac{1}{3{\cdot}9 \times 10^{10}} = 2{\cdot}6 \times 10^{-11}$$

Since this value is less than the solubility product of calcium oxalate ($2{\cdot}57 \times 10^{-9}$), the calcium oxalate will in fact dissolve completely.

If corresponding values are taken for copper sulphide, which is very insoluble—

$$\frac{[CuY^{2-}]}{[Cu^{2+}] \times [Y^{4-}]} = 2{\cdot}4 \times 10^{18}$$

and $$[Cu^{2+}] \times [S^{2-}] = \frac{1}{2{\cdot}4 \times 10^{18}}$$

This value is much greater than the solubility product of copper sulphide ($8{\cdot}5 \times 10^{-45}$). Thus ethylenediaminetetra-acetic acid will not dissolve this compound.

These values of K have an important practical significance, which will be referred to later.

The patent covering the use of iminodiacetic acid derivatives[3] specifies (*a*) at least two carboxylic acid groups per nitrogen atom, and (*b*) attachment of the carboxylic acid groups through one carbon atom to the nitrogen.

The importance of the ratio of carboxyl groups to nitrogen atoms may be illustrated by considering the reaction between ammonia and chloroacetic acid. Three compounds may be formed—

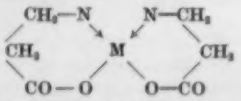
$$NH_3 + ClCH_2{\cdot}COOH \longrightarrow \underset{\text{Aminoacetic acid}}{NH_2{\cdot}CH_2{\cdot}COOH} \quad (I)$$

$$NH_3 + 2ClCH_2{\cdot}COOH \longrightarrow \underset{\text{Iminodiacetic acid}}{NH(CH_2{\cdot}COOH)_2} \quad (II)$$

$$NH_3 + 3ClCH_2{\cdot}COOH \longrightarrow \underset{\text{Nitrilotriacetic acid}}{N(CH_2{\cdot}COOH)_3} \quad (III)$$

Compounds II and III possess metal-sequestering properties, but aminoacetic acid (I) is of no value in this respect.

The importance of the position of the carboxyl groups relative to the nitrogen atom may be inferred from the formulæ of the metal complexes, in which the metal is chelated in two five-membered rings. Six-membered metal chelate rings would also be expected to be stable, so that compounds in which the carboxyl groups are separated from the nitrogen atom by two carbon atoms should also have metal-sequestering properties—



Such derivatives of iminodipropionic acid do not appear to have found any use commercially, possibly because they would be too expensive to manufacture. It is interesting to note that workers in the U.S.A. have recently compared metal derivatives of iminodiacetic acid and of iminodipropionic acid, and find the latter to give less stable complexes[4].

Of the numerous possible derivatives of iminodiacetic acid, only two have been marketed commercially, namely ethylenediaminetetra-acetic acid and nitrilotriacetic acid. The former has been more widely used, since it forms more stable complexes, thus giving better technical performance. A possible reason for the greater stability may be deduced from the formulæ of its metal complexes. The two nitrogen atoms to which the metal is co-ordinated are also joined in a third five-membered ring, giving an evidently stable and compact structure. In the case of nitrilotriacetic acid separation of the two molecules of acid with regeneration of the metal ion is possible. Nitrilotriacetic acid, however, is made from a cheaper raw material (ammonia) than ethylenediaminetetra-acetic acid (ethylenediamine), and for this reason it can compete economically with the polyphosphates for certain purposes.

Two other interesting iminodiacetic acid derivatives are those derived from diethylenetriamine

$$H_2N{\cdot}CH_2{\cdot}CH_2{\cdot}NH{\cdot}CH_2{\cdot}CH_2{\cdot}NH_2$$

and from triethylenetetramine—

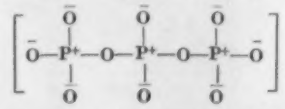
$$H_2N{\cdot}CH_2{\cdot}CH_2NH{\cdot}CH_2{\cdot}CH_2{\cdot}NH{\cdot}CH_2{\cdot}CH_2{\cdot}NH_2.$$

These are excellent products, but they do not appear to be any better than ethylenediaminetetra-acetic acid, and since the two amines are expensive (both are made from ethylenediamine), the cost of manufacture would probably be prohibitive.

The complex-forming action of the phosphates is probably similar in character to that of iminodiacetic acid derivatives. A full discussion is beyond the scope of this paper, but a comprehensive summary has been prepared by Topley[5]. For example, sodium pyrophosphate forms complexes of the type $M^{III}P_2O_7^-$ and $M^{II}P_2O_7^{2-}$ with tervalent and bivalent metals respectively, in a manner analogous to ethylenediaminetetra-acetic acid.

Another feature of the complex anion formation with the condensed phosphates is that reaction occurs with most bi- and ter-valent metals, including those, such as calcium and magnesium, not very prone to complex formation. In this respect the phosphates show the same behaviour as the organic sequestering agents.

In the case of sodium triphosphate the anion is considered to have the following structure—



Complex formation is thought to take place by interaction of the negative charges on the –O·P·O·P– chain with the positively charged metal ion. It will be seen that the reaction is similar to complex formation with iminodiacetic derivatives, where the positive charge on the metal ion is neutralised by interaction with the electrons on the nitrogen atoms.

The action of the phosphates in dissolving slightly soluble salts will be subject to similar considerations to those described for ethylenediaminetetra-acetic acid.

TEXTILE APPLICATIONS OF SEQUESTERING AGENTS

"Sodium hexametaphosphate" was introduced into Great Britain about 1932[6], and nitrilotriacetic acid and ethylenediaminetetra-acetic acid at least as early as 1938[3]. They are not, therefore, new products, but in recent years there appears to have been a greater appreciation of their possibilities. Although the three compounds have a similar mode of action, they show certain differences in behaviour which are of practical importance and which, quite apart from any question of cost, may influence the choice of reagent for a certain purpose. It is convenient to consider the practical uses of these compounds in relation to the more important metals.

(1) *Calcium and Magnesium*

These metals are present to varying extents in most natural waters and their effects are well known. Addition of sufficient sequestering agent to a hard water will reduce the concentration of metal ion to almost zero, although the amount of metal present is unchanged. The calcium and magnesium are present in a complex form and will not precipitate soap. A hard water can thus be rendered suitable for soap scouring. Where the water used is very hard it may, of course, be more economical to install a water-softening plant. In waters where the hardness is not high, scouring can often be carried out satisfactorily without softening. Even so, a certain amount of insoluble soap is formed, and this may tend to separate out in rinsing. Ethylenediaminetetra-acetic acid has in one case been found to give sufficient improvement in wool scouring to justify the increased cost, even though results prior to its introduction were considered satisfactory. "Sodium hexametaphosphate" has, of course, been used in wool scouring for a long time, and it is the practice of some dyers to wet out wool pieces in this reagent to remove any insoluble soaps. The major use of sodium triphosphate and "hexametaphosphate" is in scouring and other cleansing processes. Their usefulness in this respect depends partly on their ability to sequester calcium and magnesium. In addition they possess the property of exerting a dispersing effect on finely divided solids, and their use in cleansing processes is partly attributable to this property.

Many wool dyes can be applied successfully in waters of quite high hardness, e.g. 8–10 parts of calcium carbonate per 100,000, so that softening may not generally be necessary. A few wool dyes, however, are liable to precipitate partly or completely in hard water. A rather extreme case can be demonstrated by dyeing a medium to heavy depth of Polar Red RS (Gy) in water with a hardness of 20 parts of calcium carbonate per 100,000. The dye is partly precipitated, and the resulting dyeing rubs badly. Addition of ethylenediaminetetra-acetic acid prevents precipitation, and the dyeing has a rubbing fastness equal to that of one carried out in distilled water. In such cases the use of a sequestering agent may be more economical than installation of a water-softening plant, since only a limited number of dyes are affected.

With some wool dyes which are less sensitive to hard water, it may be found that calcium and magnesium salts cause slight development of scum on the surface of the dye liquor, even with waters of low hardness. This may be a serious matter in machine dyeing if there is any filtration effect, and a sequestering agent will assist in preventing this and giving cleaner results. Many direct cotton dyes are affected by hard water, being either precipitated or changed in dyeing properties; e.g. many Class B dyes (S.D.C. classification)[7] show the properties of Class C dyes. M. V. Campbell found that such difficulties could be avoided by the use of "sodium hexametaphosphate" in the dyebath[8].

When sequestering agents are used in dyeing or other processes involving treatment at a high temperature, it should be remembered that "sodium hexametaphosphate" gradually changes to sodium orthophosphate on boiling, thus losing its sequestering properties. The change is more rapid in the presence of acid or alkali. Iminodiacetic acid derivatives are stable to prolonged boiling.

(2) *Iron*

Machinery with iron fittings may be a source of trouble during textile processing, and some natural waters contain appreciable quantities of iron, usually in the form of ferrous bicarbonate. The most common trouble caused by iron is that it seriously affects the colour of many chrome dyes, particularly when dyed by the afterchrome method. This problem has been studied by Bird and Molloy[9], who examined ethylenediaminetetra-acetic acid, "sodium hexametaphosphate", sodium triphosphate, sodium pyrophosphate, and sodium dihydrogen pyrophosphate. They found that in the pH range 4–6 ethylenediaminetetra-acetic acid gave good protection against iron with all the dyes examined. The results obtained with "sodium hexametaphosphate" and sodium dihydrogen pyrophosphate varied with different dyes. With Eriochrome Verdone S (Gy) these two phosphates gave slightly inferior results, and with a number of dyes only ethylenediaminetetra-acetic acid gave good protection. In the case of Omega Chrome Red G (S) "sodium hexametaphosphate" gave complete, and sodium dihydrogen pyrophosphate almost complete, protection. Sodium pyrophosphate under similar pH conditions gave results similar to the acid pyrophosphate. Sodium triphosphate was found to be less effective than the other reagents examined.

Under slightly acid conditions ethylenediaminetetra-acetic acid is the most effective agent for preventing off-shade effects caused by iron. Above pH 6·0 it tends to lose its protective effect as the pH rises. Nitrilotriacetic acid, although it will form a complex with iron, is ineffective in preventing off-shade effects in chrome dyebaths.

It is difficult to make specific recommendations regarding the amount of sequestering agent for use in bulk dyeing, since this depends on the amount of iron contamination and the depth being dyed, pale colours being more seriously affected than dark ones. Bulk experience with ethylenediaminetetra-acetic acid indicates that the amount which

will be effective is less than would appear from laboratory trials.

Chrome dyes are not the only wool dyes affected by iron. J. F. Gaunt has shown that certain acid anthraquinone blues are catalytically hydrolysed by traces of iron in acid dyebaths, the amino groups of the dye being replaced by hydroxyl with a dulling and reddening of colour[10]. Ethylene diaminetetra-acetic acid, nitrilotriacetic acid, and "sodium hexametaphosphate" will prevent the change. It is interesting to note that in such cases, where there is no actual combination between iron and dye, all three sequestering agents are effective, in contrast to chrome dyes, where there is competition between the dye and the sequestering agent.

Dyeings of good wet fastness may be obtained on cellulosic fibres by the use of special ranges of direct dyes which are aftertreated with copper salts, e.g. Cuprophenyl (Gy) dyes. Since these dyes contain metal-coordinating groups, it is not surprising to find that they are affected by iron. Ethylenediaminetetra-acetic acid is effective in overcoming this trouble.

Iron stains are a frequent occurrence on textiles, and are retained tenaciously by wool. Oxalic acid is often used for removing such stains. Iron is readily removed from cellulosic fibres in this way, but in the case of wool the oxalic acid merely reduces the intensity of colour of the stain, the iron–oxalic acid complex not being readily removed by rinsing. The presence of iron after treating with oxalic acid is readily demonstrated by spotting with acidified potassium ferrocyanide On a short exposure to light the stain is liable to be restored to a grey. Tartaric and citric acids will give similar results. Phosphoric acid also will decolourise, but not remove, iron stains on wool, but there is little tendency for discoloration to occur on exposure to light. The ability of ethylenediaminetetra-acetic acid to form complexes with iron would suggest that it can remove iron stains from textiles, but it is in fact ineffective when used alone.

The addition of oxalic, phosphoric, tartaric, or citric acid to ethylenediaminetetra-acetic acid will, however, bring about complete removal of iron stains from wool. Even at 60°C. complete removal is obtained, unless staining is exceptionally heavy, when treatment at a higher temperature is necessary.

The ability of oxalic acid, etc. to bring about iron removal in conjunction with ethylenediaminetetra-acetic acid is probably related to their tendency to form complexes with iron and not due to a reduction in pH value. Sulphuric acid at similar pH values is less effective. Reducing agents, particularly sodium hydrosulphite, also assist in removal of iron stains when ethylenediaminetetra-acetic acid is used.

(3) *Copper*

The effect of copper on chrome dyes is similar to that of iron, and off-shade results due to copper can be prevented by using ethylenediaminetetra-acetic acid. Since "sodium hexametaphosphate" gives some protection against iron in chrome dyebaths, it is surprising to find that it gives little protection against copper. Sodium pyrophosphate is stated to have some protective action, but has not been investigated by the present author. An aspect of the effect of copper on chrome dyes to which attention has recently been drawn arises from the use of copper sulphate in sheep dips[11]. The amount of copper retained is said to be sufficient to affect seriously some chrome dyes when dyed in pale colours on loose wool, and ethylenediaminetetra-acetic acid effectively prevents such off-shade results. Nitrilotriacetic acid is ineffective in preventing off-shade results in chrome dyebaths resulting from the presence of copper.

The stronger sequestering action of ethylenediaminetetra-acetic acid on copper, compared with that of sodium "hexametaphosphate", is not always an advantage in favour of the former. For example, if a copper-containing direct dye is dyed in hard water, it may be necessary to use a sequestering agent. If ethylenediaminetetra-acetic acid is used, its strong affinity for copper results in removal of the metal from the dye molecule, with a change of colour and loss of fastness properties. Nitrilotriacetic acid shows a similar behaviour, whereas sodium "hexametaphosphate" appears to have much less effect. Campbell's observation that ethylenediaminetetra-acetic acid destroys the colour of certain direct dyes may be due to the fact that the dyes referred to contain copper[8].

Copper stains are not readily removed from wool by ethylenediaminetetra-acetic acid. This is probably due to the fact that such stains are usually present as copper sulphide resulting from interaction of copper and the natural sulphur in the wool. As shown previously (p. 280) copper sulphide, owing to its low solubility product, is not dissolved by ethylenediaminetetra-acetic acid. Cellulose fabrics which are rubberised have to be practically completely free from copper, and this can be accomplished by the use of ethylenediaminetetra-acetic acid. This compound has also proved useful in the linen industry, where copper gives rise to a great deal of trouble owing to its catalytic effect in bleaching.

(4) *Miscellaneous Applications*

Calcium, magnesium, iron, and copper are the metals most commonly responsible for textile faults. The action of ethylenediaminetetra-acetic acid on other metals is, however, of interest. Lead stains on wool formed by reaction of the metal and sulphur in the wool are an occasional occurrence, lead sulphide being formed. Such stains are not readily removed by sequestering agents, owing to the low solubility product of lead sulphide. An investigation in the author's laboratory of the development of stains in union dyeing showed that these were caused by lead. Addition of ethylenediaminetetra-acetic acid to the dyebath prevented their development. Sodium "hexametaphosphate" and nitrilotriacetic acid also had some protective effect.

Fabrics which have to be rubberised must be virtually free from manganese as well as copper. Ethylenediaminetetra-acetic acid will remove manganese completely.

Ethylenediaminetetra-acetic acid and nitrilotriacetic acid form complexes with chromium, and

for this reason cannot be used in the dyeing of those chrome-complex dyes which are applied from a strongly acid bath, the chromium being removed from the dye molecule and the colour of the unchromed dye being obtained. Sodium "hexametaphosphate" has less effect on these dyes. The neutral-dyeing metal-complex dyes are more resistant to sequestering agents. All the Irgalan (Gy) dyes are unaffected in colour or fastness properties. The question of using a sequestering agent with chrome-complex dyes rarely arises, since the majority are not sensitive to metals.

ANALYTICAL APPLICATIONS AND EVALUATION

No account of the uses of sequestering agents is complete without some reference to the analytical applications of ethylenediaminetetra-acetic acid. All the uses cannot be covered in the present paper, but the procedures developed by Schwarzenbach and his co-workers for water hardness determination are of particular interest in relation to textile processing. One method depends on the change in pH when the di- or tri-sodium salt reacts to form complexes with calcium or magnesium—

$$Ca^{2+} + H_2Y^{2-} \rightleftharpoons CaY^{2-} + 2H^+$$
$$Ca^{2+} + HY^{3-} \rightleftharpoons CaY^{2-} + H^+$$

The liberated hydrogen ion may be determined potentiometrically, or by titration using a suitable indicator[12].

In an alternative method, the water is titrated at pH 10 with the disodium salt using Eriochrome Black T (*C.I.* 203) as indicator[13]. The dye, which is blue in alkaline solution, forms red complexes with calcium and magnesium. During titration the calcium and then the magnesium are extracted from the metal–dye complexes, and the indicator changes from red to blue at the end-point. By using as indicator murexide (ammonium purpurate) which is not affected by magnesium, calcium hardness can be titrated separately. The Eriochrome Black T titration method can be reversed and adapted for the estimation of the active constituent in commercial samples of ethylenediaminetetra-acetic acid.

Another simple titration method depends on the fact that, in the presence of ethylenediaminetetra-acetic acid, sodium oxalate will not precipitate calcium above pH 10. In this method the product is titrated with a calcium salt, the end-point being indicated by the precipitation of calcium oxalate[14].

Polarographic methods based on the formation of the copper complexes have been worked out for ethylenediaminetetra-acetic acid and nitrilotriacetic acid[15, 16].

* * *

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Application of Radioactive Tracer Techniques to Textile Research

HOWARD J. WHITE, Jr.

Meetings of the Midlands Section held at the Gas Board Theatre, Nottingham, on 29th April 1953, Mr. A. P. Kershaw in the chair; of the West Riding Section held at the Midland Hotel, Bradford, on 30th April 1953, Mr. H. Foster in the chair; and of the Manchester Section held at the Textile Institute, Manchester, on 24th April 1953, Mr. J. Boulton in the chair

Some properties of radioactive tracers are reviewed briefly, and the advantages of their unique properties are pointed out. Several applications of tracer techniques to problems dealing with fibres are described.

It is the primary purpose of this paper to discuss the application of radioactive tracer techniques to textile research. Methods will be emphasised more than the interpretation of results. However, in order to maintain a sharp focus on textile problems, examples will be drawn, where possible, from applications pertinent to various branches of fibre research. To this end the unique characteristics of radioactive materials will be discussed briefly with emphasis on those factors which are advantages and disadvantages for tracer experiments, and then the uses to which radioactive materials have been put in the textile field will be outlined. In a few cases experimental techniques will be given in some detail to enable those unfamiliar with the field to understand the processes involved in typical experiments.

ADVANTAGES OF TRACER TECHNIQUES

The decomposition of a radioactive isotope is, as far as is known, uninfluenced by the chemical nature of its surroundings. This means that, with appropriate corrections for absorption and scattering, the amount of radiation produced in a given time is a measure of the amount of a given radioactive isotope present, independently of chemical factors such as the molecule in which the isotope is incorporated, the solvent in which it is dissolved, etc. This essential independence of the measuring technique of chemical factors is the basis of the usefulness of radioactive isotopes. Amounts can be measured accurately which are several orders of magnitude smaller than the amounts accurately measured by standard chemical or physical methods. In general, 0·01 p.p.m. and 0·01 μg. are quite generous lower limits for relative and absolute sensitivities of chemical or physical analytical methods. 0·01 μg. corresponds roughly to 10^{12} molecules as a minimum. In contrast to this, there are many cases where 10^5–10^6 radioactive atoms can be detected and measured in a short time, even if they are incorporated in much larger numbers of inactive atoms.

For consideration of the properties of radioactive materials and of counting, texts on radiochemistry should be consulted (see, for example, Friedlander and Kennedy [1]).

A corollary to this extreme sensitivity is ease in locating radioactivity in complex systems. With patience and a magnifying glass, one blue cotton fibre could be located in several million white ones. One fibre suitably tagged with a radioactive isotope could be located much more easily by scanning the fibre mass with a counter.

APPLICATION TO MECHANICAL PROBLEMS

The combination of great sensitivity with ease of location makes radioactive isotopes ideal tools for following complex mechanical processes and measuring distributions. Three examples of such applications in the fibre field can be given—

(1) Nylon yarn is oiled to improve its weaving and knitting characteristics. The efficiency of the oiling process is related to the uniformity of the distribution of the oil along the yarn. Measurement by chemical methods is obviously not feasible. However, by incorporating a component containing radioactive bromine into the oil, the uniformity of the distribution is easily followed by measuring the radiation from the bromine. This and other possible applications have been briefly reported by Trotman [2].

(2) In printing, the carrying of colour from one box to the next by the printed piece and backing cloth can lead to off-colour shades as the printing proceeds. If a box is known to be contaminated, it can be emptied and refilled; however, a method of testing for contamination is necessary. By adding a radioactive tracer, in the form of ammonium phosphate containing radioactive phosphorus, to the dye in the first box and attaching an appropriate set of Geiger counters to the second, the increase in concentration of the contaminating dye can be followed continuously and the box recharged when that concentration becomes excessive [3].

(3) In the manufacture of paper, the pulp fibres are passed through a device known as a *Bird screen* before being consolidated into the paper sheet. The Bird screen is essentially a rotary filter. On large papermaking machines there may be several Bird screens all feeding fibres into the same head box. As with most fibrous assemblies, uniform distribution is necessary for uniform quality, and a knowledge of fibre flow patterns through the system is necessary in controlling uniformity and designing new equipment. By tagging fibres fed into one Bird screen with a suitable radioactive isotope and measuring the resulting radiation on the paper made from the fibres, the flow pattern through the papermaking machine can be established in some detail. Such an experiment has been thoroughly described by Sankey, Mason, Allen, and Keating [4]. Since the experiment was run on plant equipment and the problem studied is similar to problems in carding and blending met in textile manufacturing, it may be of interest to consider some details of the method.

The radioactive isotope used was ^{131}I, which has a half-life of 8·0 days and emits both β- and γ-radiation. The less strongly attenuated γ-rays were useful in long-range qualitative measurement of activity on the paper, and the more strongly ionising β-particles in quantitative measurement. As is well known, overexposure to radiation can cause serious physical consequences, so that it was necessary to safeguard the health of operators and ultimate users of the fibres or paper. The relatively short half-life of the isotope lessened the possible time of immobilisation of the equipment in case of contamination and shortened the storage time necessary before the fibres could be reused for commercial production.

The fibres were soaked in 14% silver nitrate solution, treated with N. sodium chloride solution, and washed vigorously. Part of the chlorine was then replaced by radioactive iodine by treatment with an alkaline radioactive sodium iodide solution. Preliminary experiments showed that there was no exchange of activity between fibres and very little mechanical loss of silver iodide crystals during processing. 500 g. of groundwood fibres were tagged with 100 millicuries of ^{131}I in a 43-gal.(U.S.A.) can.

Qualitative checks on the flow of activity through the papermaking machine were made by means of counting-rate meters stationed above the moving paper. Quantitative measurements were made by means of pieces of paper folded into cylinders and a cylindrical Geiger counter. Radioautographs were used to show the fine-scale distribution of the tagged fibres within a sample of paper. At the conclusion of the experiment, 8×10^{-7} g. of radioactive iodine was distributed throughout $3\frac{1}{2}$ tons of paper having an area of 6×10^5 sq. ft.

APPLICATION TO ABSORPTION AND DIFFUSION MEASUREMENTS

Before a radioactive isotope can be used as a tracer in a given chemical process, it is necessary to enquire whether it will undergo the process to the same extent and at the same rate as would the naturally occurring isotope or mixture of isotopes. The answer is usually "yes" with respect to both equilibrium and rate. The differences in behaviour are related to the fractional changes in molecular weight resulting from the substitution of a radioactive isotope for a natural isotope. For elements beyond the first row in the periodic table, the differences are negligible for most practical purposes, although isotopes actually can be separated by techniques such as diffusion through membranes or thermal diffusion. For elements in the first row of the periodic table, appreciable effects are possible. As an example, Lindsay, Bourne, and Thode [5] found that the rupture of a ^{12}C–^{12}C bond in the decarboxylation of malonic acid was 2% faster than the rupture of a ^{12}C–^{13}C bond. Others have found different percentages for different reactions. The extent of the influence of isotope mass on reaction rates is not clear but is certainly fairly small except for the lightest isotopes. Since, with the exceptions noted above, radioactive isotopes can be expected to have the same chemical properties as the naturally occurring isotopes of the same element, their unique radiative properties can be used to follow various chemical processes.

The sensitivity of tracer techniques makes it possible to think of using them to follow chemical processes occurring within a single fibre. These include the rate of penetration of the fibre, equilibrium uptake under various conditions, and, in some cases, the rate of chemical reaction between active groups within the fibre and an absorbed reagent. These steps are fundamental to all dyeing, finishing, and bleaching operations.

Much work has already been done with samples composed of many fibres. However, there are two problems arising from the use of such samples that are difficult to circumvent. If the sample is removed from the treating solution, it is difficult to distinguish between absorbed solution and solution adhering to the surface of the sample. If the sample is not removed from the solution, it is necessary that one component of the solution be absorbed differentially, so that the change in concentration of the solution can be measured. Furthermore, the rate of penetration of absorbate will usually be a function of the complex geometry of the sample as well as being dependent on diffusion processes within the fibres.

These difficulties can be minimised when single fibres are used as samples. The following technique has been found to be satisfactory with human hair fibres and has been applied to studies of the absorption of alkali-metal bromides by the hair [6]. These experiments were done as part of a study of the fundamentals of dyeing, and both the equilibrium uptake of salt and the rates of absorption and desorption were of interest.

Hair fibres, 10 cm. long, were conditioned and weighed on a microbalance. The radioactive bromide in the form of a potassium bromide solution was passed through an ion-exchange resin on the acid cycle to form hydrobromic acid, which was titrated with the appropriate alkali-metal hydroxide. The fibres were then immersed in the bromide solution, removed, and washed. Supplementary experiments showed that a 2-sec. wash in ethyl alcohol was satisfactory for removing adhering liquid without removing absorbed salt.

The fibres were coiled on stainless-steel discs, and their radioactivity was measured by means of a flow counter, similar in principle to a Geiger counter. A similar disc on which 2 c.c. of a solution of known concentration had been evaporated was used as a standard. It was necessary to correct for absorption of radiation within the sample. This was done by applying a correction coefficient determined from other experiments in which the fibres were dissolved and spread on the discs in very thin films which absorbed negligible amounts of radiation. The hair samples averaged roughly 200 μg. in weight, and amounts of salt as small as 2×10^{-7} g. (0·2 μg.) were measured. In other experiments [7], an acid dye for wool (*C.I.* 1066) containing radioactive sulphur was synthesised and used in absorption measurements.

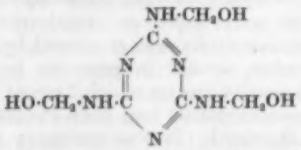
Wall and Swoboda [8] have used radioactive sodium to determine the amount of sodium hydroxide taken up by nylon from solutions which were

too concentrated to be analysed by the conventional change-of-titre technique. The nylon samples were dissolved in 88% phenol solution and counted in solution. A correction was made for adhering liquid.

Studies on the absorption and diffusion of hydrobromic acid in horn keratin have been made by Wright [9]. Diffusion coefficients and ionic mobilities were measured, radioactive bromine being used as the tracer. In these experiments, use was made of an experimental measurement which can be made only with radioactive materials. This is the determination of the self-diffusion coefficient.

In polymeric systems it is quite common that experimental differential diffusion coefficients depend on the concentration of diffusant. This makes interpretation of diffusion experiments much more difficult. Furthermore, if diffusion across a chemical concentration gradient in a multicomponent system is followed, the resulting change is a function of several interdependent diffusion processes. However, if a solution containing one radioactive component is placed in contact with another solution, identical except for the fact that none of its components are radioactive, there are no chemical concentration gradients. The flow process is an exchange of radioactive molecules or ions for inactive molecules or ions. This process is known as *self-diffusion*. The self-diffusion coefficient is directly related to the local Brownian motion of the component in question, and this in turn is quite revealing with respect to local molecular order. Thus Wright was able to show, from ionic mobility measurements, that for horn keratin at equilibrium with solutions of low acid concentration, most of the current was carried by bromide ions, with the hydrogen ions strongly held by the film. At high acid concentrations the current carried by the hydrogen ions increased rapidly, indicating the presence of a less strongly held fraction.

Dixon, Christopher, and Salley [10] used the self-diffusion principle to study a solution similar to many found in the washing, dyeing, and resin-treating of textiles. Trimethylolmelamine, which has the structural formula—



polymerises in aqueous acid solutions; the polymer is useful for imparting wet strength to paper. The monomer is basic and binds hydrogen ions. As the polymerisation proceeds, the positive charge on the polymer unit (which would otherwise become prohibitively large) is reduced by the loss of hydrogen ions and the binding of counter-ions, usually chloride ions. A knowledge of the number of chloride ions bound is necessary to determine the essential properties of the polymer solution. By comparing the ionic mobilities of solutions of the polymer with those of sodium chloride solutions of the same chloride ion concentration, the proportion of chloride ion bound to the polymer could be determined. Actually, radioactive bromide ions were used as a tracer for the chloride ions, a correction being made to account for the difference in mobility between the two ions.

APPLICATION TO DETERMINATION OF POLYMER STRUCTURE AND REACTION MECHANISMS

An interesting application of tracer techniques has been made by Levy and Slobodiansky [11] using methods developed by Keston and Udenfriend [12]. A complete, or partial, hydrolysate of a protein, in this case silk, is treated with *p*-iodobenzenesulphonyl chloride containing radioactive iodine. This reagent combines quantitatively with amine end-groups. The total radioactivity of the hydrolysate is then measured, and known amounts of various inactive derivatives suspected of being present are then added. The various derivatives are then isolated and purified, no particular attempt being made to obtain a quantitative recovery, since the weight and the activity of the final sample are adequate to determine the amount of the given species initially present. The ratio of the final specific activity to the activity which would be present if every molecule contained a radioactive atom (obtainable from the weight and the half-life of the radioactive isotope used) is $w/(w + W)$, where w is the weight of the component present in the unknown and W is the weight of carrier added.

Levy and Slobodiansky prepared carriers for the partial hydrolysates— glycylalanine, alanylglycine, and glycylglycine. Analysis of two hydrolysates showed that under the conditions of hydrolysis alanylglycine was five times as prevalent as glycylalanine, which in turn was much more prevalent than glycylglycine. This suggests that the repeating unit alanylglycine occurs frequently in silk and is resistant to the method of hydrolysis used. More work of this kind should give more information on the order of amino acids in silk, and it would seem worth while to do similar experiments on wool.

One further use for tracers should be mentioned, although apparently it has not yet been applied to textile problems. This is in evaluating reaction mechanisms. As an example, consider the reaction—

$$K_2CO_3 + 2NH_2{\cdot}C^*O{\cdot}NH_2 \rightarrow 2KC^*NO + CO_2 + H_2O + 2NH_3$$

where the asterisk is used to denote radioactive carbon.

Presumably, alternative mechanisms for the reaction could be proposed in which either carbon on the left-hand side becomes part of the cyanate on the right. However, the use of urea tagged with radioactive carbon and the appearance of the activity in the cyanate eliminated mechanisms in which the carbon from the potassium carbonate remains in the cyanate. This example is taken from the work of Williams and Ronzio [13] as reported by Ropp and Neville [14], who give an excellent review of the uses of isotopic carbon covering the years 1949–1951. As has been mentioned previously work with carbon, particularly rate work, is subject to possible error from isotope effects.

CONCLUSIONS

Radioactive tracers have been used already in a number of applications in the field of fibre research

where the results either could not be duplicated in any other way or only by use of very elaborate and less accurate methods. They are usually easy to use, the biggest problems being synthesis of an appropriate tagged compound and avoidance of health and contamination hazards when large-scale experiments are done. Finally, it might be mentioned that the catalogue of Oak Ridge National Laboratory for July 1952 lists separated radioactive isotopes for 54 elements, the most important omissions, from an organic chemist's point of view, being oxygen and nitrogen. A similar catalogue is available from the Atomic Energy Research Establishment at Harwell.

* * *

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Discussion

MANCHESTER SECTION

Mr. A. S. FERN: Can the tracer technique be used to assess the penetration and location of a dye (or other penetrant) in a fibre or filament cross-section?

Dr. WHITE: The logical approach would be use of radioautographic techniques with fibre cross-sections. There is a radioautographic technique in the literature for which a resolution of 1μ. is claimed [15]. This resolution would be adequate for most textile problems. However, the process is a wet process. We have tried it, and have found that migration of absorbed materials occurs during exposure of the film, thus making the method useless for our purposes.

Mr. D. A. GARRETT: In an attempt to use tracer technique to study distribution of a dye across a fibre cross-section, could not the resolving power be considerably increased by cutting a thin wedge of the fibre and scanning along the wedge by a photographic plate method?

Dr. WHITE: The resolving power would certainly be increased in this way, but I doubt whether it would be increased enough to produce adequate resolution with the dry radioautographic techniques now available. A wet technique has the disadvantage mentioned above.

Mr. FERN: Sulphonated dyes can readily be obtained in a radioactive form by sulphonating with sulphuric acid containing radioactive sulphur. How would radioactive vat and disperse dyes be prepared easily?

Dr. WHITE: There is no general answer to this question. Radioactive carbon itself and a number of common reagents in which radioactive carbon is incorporated are available. Thus a radioactive form of any given vat or disperse dye could be made using carbon. The ease of preparation would depend on the particular dye involved.

Mr. GARRETT: Since the synthesis of a dye from components incorporating activated atoms would be a lengthy process, could the dye itself be activated by introduction into the atomic pile?

Dr. WHITE: In principle the success of such a process depends on the composition of the dye. For example, bromine atoms can be activated in the pile; in other cases parent and daughter atoms have different atomic numbers. Practically speaking, undesirable secondary activation reactions and decomposition of the dye would probably make the process unfeasible.

Mr. H. R. HADFIELD: Is there a possibility of using unstable isotopes for investigating the mechanism of development of the sulphuric esters of leuco vat dyes?

Dr. WHITE: I would think so. Again the feasibility depends on the dye chosen, the position in the dye at which the radioactive isotope is desired, and the ingenuity of the worker.

Mr. F. FARRINGTON: What are the effects on various textiles of insertion in the atomic pile?

Dr. WHITE: Only preliminary results are known. Various reactions are initiated by the radiation; e.g. with some polymers cross-linking occurs. Eventually destruction of the fibres will probably occur (see the work of Charlesby and co-workers [16]).

Mr. F. FARRINGTON: Can such radiation be continuously measured over a moving fabric surface?

Dr. WHITE: Yes. Counting-rate meters are available, capable of measuring the activity on a moving sample. A variation of this in which an inactive sheet moves over a fixed source is used in certain gauges devised to measure the thickness of moving sheets of material. The counting-rate meters do not give the accuracy obtainable with a Geiger counter and scalar, however.

ANON.: Could continuous recording be effected by connecting the anode of the Geiger tube to the grid circuit of an electrometer valve, and using a suitable amplifying system with a cathode-ray oscilloscope as the recording device?

Dr. WHITE: I think this could be done. I do not know what advantages, if any, would be obtained.

COMMUNICATION

A Laboratory Apparatus for Detergency Studies

W. A. STRAW

An apparatus is described for providing controlled mechanical agitation of chopped fibre in detergent liquors on a laboratory scale, employing relatively low liquor : fibre ratios. Thermostatic temperature control is provided, and by means of calibration graphs, temperature–time conditions can be selected to follow industrial practice. An estimate is given of the accuracy of speed and temperature control.

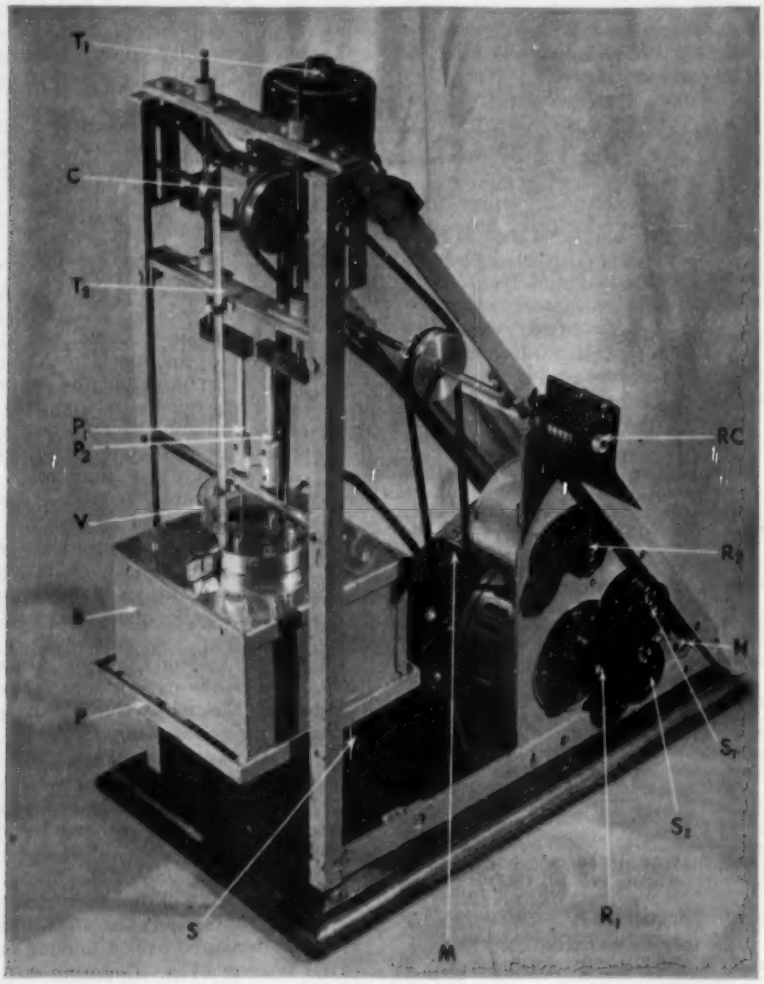
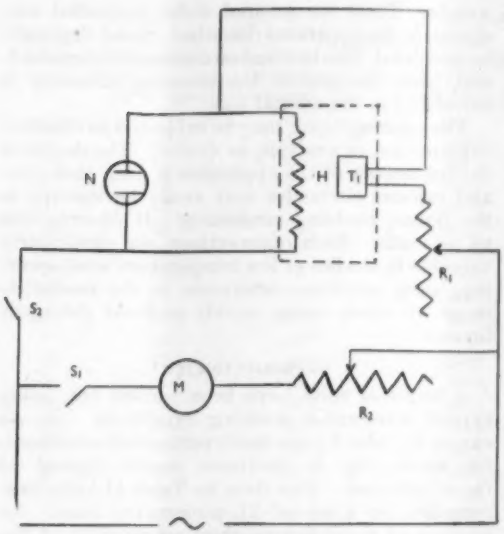


FIG. 1

Powney and Feuell [1] have described an improved technique for detergency evaluation, applicable e.g. in the routine examination of commercial products or for studying the physicochemical aspects of detergent action. These authors describe an experimental washing machine in which chopped fibre is agitated in a detergent liquor by means of a reciprocating perforated plunger.

Harris [2] surveys recently developed techniques for detergency evaluation, and mentions the use of the Detergency Comparator [3], a miniature dolly washer scaled down from a mill machine, and also the Deter-Meter [4] and Terg-O-Tometer [5], which are based on the principles of the power (falling-load) and home (agitator) washers respectively. The A.A.T.C.C. Launderometer [6] and the S.D.C. wash-wheel [7] both utilise rubber or steel balls inside the containers for providing mechanical agitation by impact. Adjustable squeeze rollers are employed in the miniature yarn-scouring apparatus described by Higgins [8] for studying continuous methods of wool scouring (on yarn or on raw wool).



R_1 500 ohms, 100 watts (Berco)
R_2 100 ohms, 50 watts (Berco)
H 100 watts
T_1 TS 2 Adjustable bimetal "thermostat", stem type, normally closed contacts (Sunvic)
M Fractional-h.p. motor, 100 r.p.m. (Fracmo Ltd.)
N Neon lamp

A.C. mains 230 v.

FIG. 2

Whichever form of machine is chosen, it is of paramount importance that in any comparative studies the amount of work done during the actual washing operation shall be controlled. Bacon and Smith [9] have shown the importance of this in connection with soil removal, whilst the influence of mechanical action in experimental scouring studies on wool has been shown by Dickinson and Palmer [10].

The author has designed and used with success a new form of laboratory scouring apparatus, with which it has been found possible to work with a much lower liquor:fibre weight ratio than has hitherto been the case with single-plunger forms. This feature is particularly advantageous when it is desired to simulate industrial scouring conditions employing liquor : goods ratios of the order of 18 or 20 : 1, as for instance in the scouring of knitted wool goods. Whilst it is convenient to process fibres in the form of chopped yarn, small squares of knitted fabrics can be accommodated as an alternative. Thus both for laboratory studies of commercial processes and for more fundamental research work in connection with the effects of scouring upon the structure of hosiery yarns and fabrics, it has been convenient to standardise arbitrarily a working ratio of 20 : 1.

DESCRIPTION

The photograph (Fig. 1) gives a general view of the assembly. It consists essentially of two circular stainless-steel paddles P_1, P_2 (approx. 1 inch in diameter), made to reciprocate vertically by means of a crank mechanism C linked through a system of triple pulleys to a series-wound fractional-h.p. motor M. The displacement of the paddles can be varied from 3 mm. to 45 mm. by adjusting the setting of the crank mechanism. A cylindrical vessel V, e.g. a 250-ml. squat Pyrex beaker, is clamped rigidly in position by a spring-clip on the heat-insulated box B. This merely contains an electric heating mantle shaped to surround the base and lower side of the beaker. This unit is maintained on a separate platform P which can be adjusted to the required height on the support S, and is clamped with a thumbscrew. R_2 is a rheostat in series with the motor, which, together with the variable-ratio pulley drive, enables the speed of agitation of the paddles to be varied between 50 and 250 strokes per minute. A revolution counter RC is incorporated. Switches S_1 and S_2 control the motor and heating circuits respectively. Rheostat R_1 controls the energy input for the heating mantle. The lower part of the thermoregulator T_1 is normally immersed in the liquor contained in the vessel V. A neon lamp N connected in parallel with the heating element indicates when the heating circuit is in use. A mercury-in-glass thermometer T_2 is clamped on the frame, so that a visual temperature check may be made at any time. The circuit diagram is shown in Fig. 2.

OPERATION

Calibration graphs have been prepared so that the time–temperature relationship and temperature conditions may be regulated as desired.

The sequence of operations is as follows—

1. From Calibration Graph I (Fig. 3), Rheostat R_1 is set to give the required rate of heating.
2. From Calibration Graph II (Fig. 4) the thermostat T_1 is set to give the required controlled temperature.

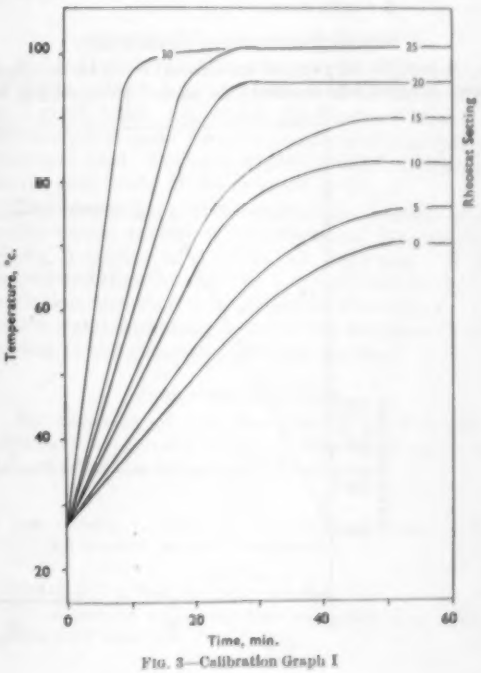


FIG. 3—Calibration Graph I

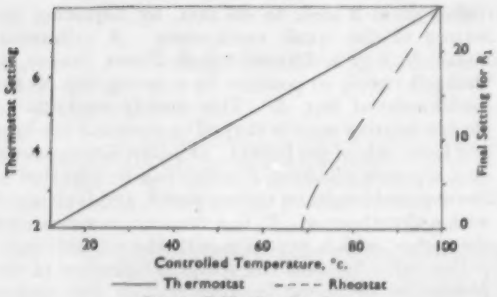


FIG. 4—Calibration Graph II

3. Agitation is begun by switching on S_1, controlling with R_2.
4. With the appropriate quantity of detergent liquor (150 ml.) in V, heating is commenced by switching on S_2.
5. When the desired initial temperature is reached, the standard load (7·5 g.) of chopped fibre is added. At this stage the timing of the actual scouring operation is commenced.
6. The speed of the paddles may now be checked.
7. When the temperature of the liquor is within 3°c. of the controlled temperature it is desired to maintain, R_1 is reset according to the rheostat curve of Calibration Graph II (Fig. 4).
8. At the end of the scouring cycle, S_1 and S_2 are switched off. The fibre-mass is separated from the liquor by filtration on a sintered-glass Büchner funnel, when it may be rinsed as desired before drying in a warm oven.

USE IN DETERGENCY EVALUATION

A sample of yarn of known oil content is taken and divided into several lots, each 7·50 ± 0·02 g. in weight. These are scoured under controlled conditions in the apparatus described, rinsed thoroughly, and dried. The residual oil content is determined, and from the results the scouring efficiency is calculated in the normal way.

The scouring liquor may be subjected to chemical examination or analysis, as desired. The design of the framework of the apparatus is such that glass and calomel electrodes may easily be inserted in the liquor, enabling continuous pH observations to be made. Such observations are particularly valuable in studies of low-temperature wool scouring, using non-ionic detergents in the isoelectric range, or when using weakly buffered detergent liquors.

REPRODUCIBILITY

A series of tests have been carried out, using typical commercial scouring conditions. As an example, Table I gives the experimental conditions, for which Fig. 5 illustrates results typical of those obtained. The data in Table II have been compiled for a set of 21 consecutive runs. An analysis of these figures gives an estimate of the reproducibility of behaviour of the machine.

TABLE I

DETERGENT LIQUOR	Composition ...	5 g. Pure Na oleate + 3 g. Na_2CO_3 (A.R.) per litre
	Quantity ...	150 ml.
LOAD	Fibre	Chopped worsted yarn (approx. 1-in. lengths)
	Quantity ...	7·50 g.
TEMPERATURE	Initial temp. ...	40°c.
AND TIME	Heating period	Raise to 60°c. in 10 min.
CONDITIONS	Steady state ...	Hold at 60°c. for 20 min.
	Total time ...	30 min.
PADDLES	Speed	Nominal 100 strokes/min. each
	Stroke	34 mm.
Temp. and revolution counter readings		At 2-min. intervals

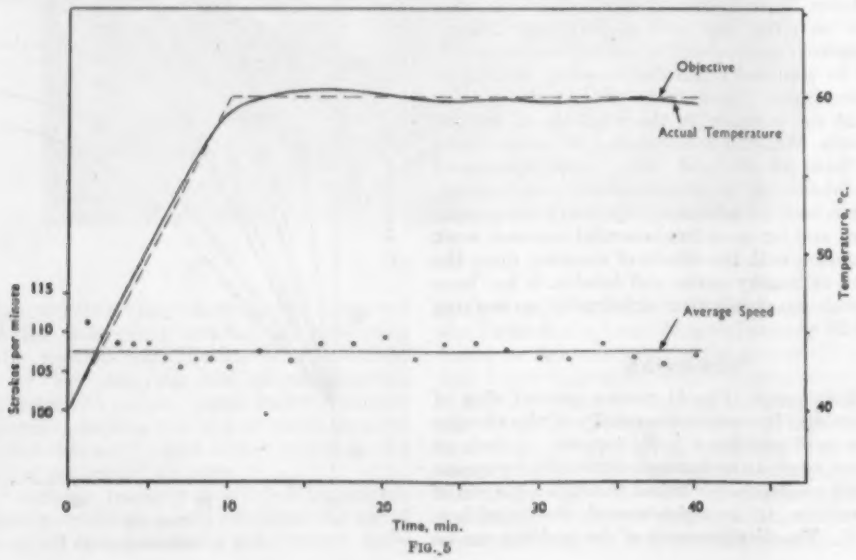


FIG. 5

TABLE II

Serial Letter	Temperature Control: Warming Period (40–60°C.) Time to reach 60°C. (min.)	Warming Period (40–60°C.) Max. Temp. attained (°C.)	Steady State (60°C.) Average Temp. during Steady Run (°C.)	Steady State (60°C.) Range of Temp. during Steady Run (°C.)	Speed Control (strokes/min.) (Nominal 100–105) Average	Speed Control (strokes/min.) (Nominal 100–105) Standard Deviation
A	10·5	61	58·2	1·4	104·8	1·7
B	11	61·2	58·1	1·2	105·4	1·9
C	13	64	59·0	0·0	106·5	2·5
D	12	61	58·8	0·5	102·0	1·6
E	12	60	58·1	0·6	100·6	1·6
F	10·5	63	58·9	1·0	104·9	1·5
G	10·5	63	59·9	0·5	107·0	1·5
H	13	61·2	60·3	0·6	105·4	2·4
I	13·5	63·2	61·0	0·8	107·3	2·4
J	12	61·2	60·6	1·1	107·2	1·6
K	15	60·2	58·9	1·3	100·5	1·8
L	14·5	62	60·2	2·0	105·8	2·3
M	12	61·5	60·9	1·4	102·4	1·6
N	12·5	62	60·5	1·4	104·2	1·5
O	11	63	61·1	1·5	105·2	1·7
P	11·5	61·2	60·6	1·6	105·0	2·9
Q	13	60·8	58·3	1·0	103·0	1·2
R	12	60·1	58·4	1·1	100·7	1·8
S	13·5	61·4	58·1	1·5	99·1	2·9
T	10·5	61	57·4	0·9	101·2	1·7
U	14	60	58·1	0·6	101·0	2·3
Average ...	12·3	61·5	59·3	1·0	103·8	2·0
Standard deviation	1·4	1·1	1·2	—	2·5	0·6
95% Limits (±)	2·8	2·4	2·5	—	5·3	1·1

Temperature–Time Relationship

It will be seen from Table II that for a temperature objective of 60°C. the average maximum temperature attained was 61·5°C., and the average steady value 59·3°C. The standard deviations of these two variables are of the same order, and it may be calculated with a good degree of confidence that the maximum temperature will be within limits of ± 2·5°C. of the objective, and that during the steady state the overall range of temperature is not likely to exceed 1·0°C.

It is desirable here to point out that these limits make allowance for—

(i) Personal errors on the part of the operative in setting T_1 and adjusting R_1 for every run; and

(ii) The temperature differential[11] characteristic of the system of thermostat control employed. This is the small but finite range of temperature obtained around the controlled value (60°C. in the present example) at a given thermostat setting, when steady conditions of hunting are achieved.

The magnitude of the temperature differential is influenced by the rate of heating, and the manner in which it is controlled. The heat capacity of the whole system, and of the thermostat unit in particular, together with the heat energy transfer processes operating, have a cumulative effect on the response of the temperature control unit.

Temperature sensitivity could be enhanced by lagging the exposed column of the thermostat, or perhaps by incorporating an electronic relay system. However, for present requirements the control achieved has been adequate. It is of interest to record that the temperature readings taken during the preparation of the calibration graph I (Fig. 3) for controlled temperatures between 30° and 75°C. showed a variation between ± 0·5° and ± 1·0°C.

Control of Mechanical Agitation

Table II shows that the average rate of operation of the paddles may be expected to be within ± 5 % of a given value, e.g. within the limits of 105 ± 5 strokes per minute, for each paddle working with a standard load. During a single run the variation is rather less, being of the order of ± 1%.

The motor drive at present in use consists of a series-wound geared unit of fractional h.p. rating, giving a torque of 4 in.–lb. at 100 r.p.m. It is considered that the value of ± 5% for stroke speed variation includes uncontrollable fluctuations in mains input and changes due to the heating effects arising in the rheostat R_2 during its use.

Detergency Evaluation

An estimate of the reproducibility of results obtained in investigations of this kind may be gained from the following typical data—

YARN

1-in. lengths, cut from unroved tubular knitted fabric; 2/24s count, worsted, 64s quality.

SAMPLES

7·50 ± 0·02 g. lots, randomly selected.

Oil content (by petroleum ether extraction on six lots): 3·07, 3·13, 3·04, 2·64, 2·54, 2·62%.
Mean = 2·84%

SCOURING CONDITIONS

5·0 g. sodium oleate + 3·0 g. sodium carbonate (anhydrous) per litre.

Liquor ratio	20 : 1
Initial temperature ...	40°C.
Temperature rise ...	1°C. per minute up to 60°C., then steady at 60°C.
Duration	Up to 30 min.
Paddle speed	105 strokes per minute
Paddle stroke	34 mm.

To study the efficiency of oil removal over the whole period, scouring was performed over 2 min., 4 min., etc. with a final run of 26 min. Table III gives the results obtained, calculated in terms of—

p_o = mean % oil content of yarn used (= 2·84%)
p_f = % oil content of scoured wool

$$\text{Scouring efficiency} = \frac{(p_o - p_f)}{p_o} \times 100\ \%$$

for each experimental run. The mean percentage efficiencies are given for each time of scouring.

It will be observed that, under the particular conditions employed, scouring efficiency is very good, even for short periods of time. Statistical analysis indicates that there is a significant difference between the mean efficiencies for different scouring times. In general, efficiency increases with time, but the rate of increase is small.

reproducibility calculated in terms of 95% confidence limits is very encouraging. Table IV shows these limits for duplicate runs up to $n = 6$. Thus for any standardised working conditions the mean of three or four repeated runs should be correct to within $\pm$ 1%.

A comparison between three typical scouring processes yielded the results given in Table V. In this case, no statistically significant differences were found between the mean percentage efficiencies. Data for the soap and soda scour are taken from the 20-min. runs quoted in Table III. Bontex (Industrial Soaps Ltd.) is a proprietary mixed nonionic and anionic detergent. The results indicate that any of the three scouring processes would be expected to prove satisfactory from the point of view of oil removal, when scaled up for commercial operation. Such a consideration can apply only to the particular oil–fibre system under test. In the experiments so far conducted, the matter (associated with the wool yarn) which is extractable with petroleum ether has been known to consist of worsted combing oil (Class A), together with knitting-machine lubricant of the mineral–fatty glyceride type.

TABLE III

Scouring Efficiency (%)

Scouring time, min. ...		2	4	6	8	10	16	20	26
Duplicate runs	1 ...	94·4	95·4	99·1	97·7	99·5	99·0	99·8	97·7
	2	94·1	93·6	97·9	98·1	96·7	99·3	99·4	99·7
	3	97·8	92·2	99·3	98·1	98·3	98·9	99·7	—
	4	—	—	—	—	99·8	99·3	99·4	—
Mean		95·4	93·7	98·8	98·0	98·6	99·1	99·6	98·7
95% Limits		±1·2	±1·2	±1·2	±1·2	±1·1	±1·1	±1·1	±1·5

TABLE IV

No. of test runs	1	2	3	4	5	6
95% Limits on mean (twice standard error) ...	±2·1	±1·5	±1·2	±1·1	±1·0	±0·9

TABLE V

Scouring liquor (per litre)		5 g. Na oleate + 3 g. Na_2CO_3	2·5 g. Bontex + 2 g. Na_2CO_3	2·5 g. Bontex + 5 g. NaCl
Temperature–time relationship ...		Enter at 40°C.; raise to 60°C. in 20 min.	50°C. for 20 min.	60°C. for 20 min.
Scouring efficiency, %	1	99·8	99·5	98·7
	2	99·4	99·7	99·5
	3	99·7	99·9	97·9
	4	99·4	—	—
Mean efficiency, %		99·6	99·7	98·7
95% Limits		1·1	±1·2	±1·2

The regression of efficiency on time was found to be significant, and the regression equation calculated from the data in Table III is of the form—

$$E\% = 0{\cdot}18\,t + 95{\cdot}80$$

(t = time of scouring in minutes). This approaches a theoretical 100% when $t = 23{\cdot}5$ min. It is felt that the small differences found with increasing scouring time are too small to be capable of interpretation in commercial practice. The statistical analysis of duplicate runs, however, shows that

The author wishes to acknowledge the helpful assistance given by Mr. G. E. Stokes in constructing the apparatus, and to express his thanks to the Director and the Council of The Hosiery and Allied Trades Research Association for permission to publish this paper.

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(*Received 12th January 1954*)

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Notes

President of the Society

At a special meeting of the Council held on Friday, 2nd July 1954, it was unanimously resolved that Mr. Fred Smith, F.S.D.C., be nominated President of the Society, to succeed the late Mr. F. L. Goodall.

Proceedings of the Council

At meetings of the Council held in the Board Room of the Dyers and Finishers Association, Commerce House, Bradford 1, on 14th April and 12th May 1954, the proceedings included the following items of interest—

DYERS AND FINISHERS ASSOCIATION—The President expressed the deep gratitude of the Council to the Board of the Association for making available their board room for this and future meetings of the Council.

PRESIDENT-ELECT—The President reported that Mr. Clifford Paine had accepted Council's nomination for election as President-elect in 1955.

RETIRING MEMBERS OF COUNCIL—The President extended his thanks to retiring Vice-presidents and Ordinary Members of Council (cf. p. 275), in particular to Mr. G. M. Williams, Vice-chairman of Council.

Mr. A. MURRAY—The President extended a warm welcome to Mr. Alex Murray, a member of the Committee of the New South Wales Section of the Society of Dyers and Colourists of Australia.

ELECTION OF CHAIRMAN—Mr. F. L. Goodall (*President of the Society*) was elected Chairman of the Council for 1954–1955.

NEW MEMBERS OF COUNCIL—The President extended a cordial welcome to new members of the Council (cf. J.S.D.C., **70**, 275 (July 1954)). It was reported that the following had recently become *ex officio* members of the Council—

Mr. J. Calvert (*Chairman, Huddersfield Section*)

Mr. H. R. Hadfield (*Honorary Secretary, Manchester Section*)

Mr. R. A. McFarlane (*Chairman, London Section*)

Mr. R. A. Peel (*Chairman, Scottish Section*)

Mr. H. W. Taylor (*Honorary Secretary, London Section*).

ELECTION OF VICE-CHAIRMAN—Mr. G. S. J. White was elected Vice-chairman of the Council for 1954–1955.

MEMBERSHIP OF COMMITTEES—The membership of committees of the Society was reviewed. Messrs. A. P. Kershaw, H. Foster, and J. W. Reidy were elected to the Finance Committee; and Messrs. S. R. Cockett, R. A. Peel, and A. Thomson to the Historical Records Committee. Mr. H. Hampson and Mr. F. Jordinson were nominated to replace Mr. J. Boulton and Mr. W. A. Edwards on the Worshipful Company of Dyers Medal and Society's Medals Committees respectively, the latter members being due to retire in order of seniority.

HONORARY PATENT AGENT—It was resolved to invite Mr. L. E. Jones to serve as Honorary Patent Agent for the ensuing year.

REPRESENTATION ON EXTERNAL BODIES—The representation of the Society on external bodies was reviewed for the ensuing year.

MEMBERSHIP—Thirty-eight applications for ordinary membership and six for junior membership were approved.

Meetings of Council and Committees

June

Council—16th
Finance—16th
Publications—22nd
Colour Index Editorial Panel—23rd
Terms and Definitions—25th
Diploma Executive Subcommittee—1st
Perkin Centenary Organising—18th

A.G.M. and Dinner 1955

Arrangements have been made to hold the Seventy-first Annual General Meeting and Dinner on Friday, 1st April 1955, at the Queens Hotel, Leeds.

Perkin Medal

Arrangements have been made for the award of the Perkin Medal to Dr. Arthur Zitscher to be made in the Large Chemistry Lecture Theatre, University of Manchester, on Friday, 15th October 1954.

The note already published (J.S.D.C., **70**, 156 (April 1954)) refers to "the discovery of the arylamides of 2:3-hydroxynaphthoic acid". Dr. Zitscher, of course, was not concerned in the discovery of the arylamides themselves but in that of the colouring matters based on them, so that the citation should read . . . *for his work leading to the discovery of the new class of azoic dyes, based on the arylamides of o-hydroxycarboxylic acids.*

Twelfth Mercer Lecture

The Twelfth Mercer Lecture will deal with *Textile Finishing* and the lecturer will be Dr. G. Landells. Arrangements have been made to hold the lecture at the Midland Hotel, Bradford, on Thursday, 17th March 1955.

Annual Meeting of Chairmen and Honorary Secretaries of Sections 1954

This meeting was held at the Midland Hotel, Manchester 2, on 1st May 1954, under the chairmanship of Mr. H. Jennison (Honorary Treasurer of the Society). Topics which were discussed included suggested subjects for lectures in the 1954–1955 session and the position in the *Journal* of notices of forthcoming meetings of Sections.

Diplomas Scheme
Fees to be paid by Candidates

The following fees are payable by candidates for the Society's Diplomas—

ASSOCIATESHIP—

£3 3s. 0d. if the whole examination is taken at once

£2 2s. 0d. for each part of the examination taken separately

FELLOWSHIP—

£5 5s. 0d. for a candidate holding the Associateship

£10 10s. 0d. for a candidate not holding the Associateship.

These will be the only fees payable by a candidate, but should an application be unsuccessful the same fees will again become payable for any subsequent application.

I.S.O. Conference on Colour Fastness at Scarborough

The third meeting of the Colour Fastness Subcommittee ISO/TC 8/SC 1 of the International Standards Organisation was held at Scarborough on 4-7th June 1954, immediately prior to the Joint Conference of the Textile Institute and the Society, and was attended by 41 delegates from 13 countries. The arrangements for the meeting were made by the British Standards Institution and the Society through its Fastness Tests Co-ordinating Committee. The countries represented were—

Belgium	Netherlands
Canada	Norway
Czechoslovakia	Sweden
Denmark	Switzerland
France	United Kingdom
Germany	United States of America
India	

Members of the Society present were—

Dr. P. W. Cunliffe	Mr. J. G. Grundy
Mr. G. B. Angus	Mr. K. McLaren
Mr. J. Barritt	Mr. J. V. Summersgill
Mr. J. Boulton	Mr. G. S. J. White
Mr. G. G. Bradshaw	Mr. E. Wilson

After an opening address of welcome on behalf of the two organisations by Dr. Cunliffe (Chairman of the F.T.C.C.), Mr. Barritt took the chair at this and subsequent meetings.

The conference first directed its attention to the range of tests on which the subcommittee had been working since its first meeting (Bournemouth 1951). These tests had been subsequently discussed in detail at the second meeting (New York 1952), and were accepted as Tentative Tests, which were published in the *Journal* in 1953 (**69**, 409). These refer to the following subjects—

General principles	Mercerising
Grey scale for evaluating change in colour	Metals in dyebath: chromium salts
Grey scale for evaluating staining	Metals in dyebath: iron and copper
Acid spotting	Organic solvents
Alkali spotting	Potting
Bleaching, hypochlorite	Pressing: hot pressing
Bleaching, peroxide	Rubbing
Carbonising, aluminium chloride	Sea water
	Soda boiling
Carbonising, sulphuric acid	Stoving
	Washing (at 40° C.)
Chlorination, acid	Water
Light, daylight	Water spotting

After further examination these testing methods and methods of assessment were accepted with only minor alterations to a few of them.

After the necessary but purely formal ratification by the main Technical Committee on Textiles (ISO/TC 38) and the Council of I.S.O., these tests will become I.S.O. recommendations and should eventually be established as national standards in each of the 33 member countries. The final version of these tests will be published in a future issue of the *Journal* and will also be adopted as a British Standard.

The conference then proceeded to examine a further series of tests, most of which had been submitted by the F.T.C.C. Ten of these were accepted as Tentative International Standards, viz.—

Acid planking	Milling, acid milling
Chlorinated water	Milling, alkaline milling
Bleaching, sodium chlorite	Steaming
Cross-dyeing	Sublimation
Decatising	Washing at 60° C.
Degumming	Washing at 95° C.

whilst two tests—Perspiration and Gas Fume fading—were deferred for later consideration.

P W C
K McL

Annual Golf Meeting

The Society's Second Annual Golf Competition (cf. J.S.D.C., **70**, 270 (July 1954)), held on Sunday, 30th May 1954, on the course of the Cavendish Club, Buxton, was a great success. The number of competitors was eighteen, and it is very much hoped that the entry for the third annual meeting, to be held in 1955, will be considerably greater.

The game was followed by a high tea, at which Mr. F. L. Goodall (*President of the Society*) presented the trophy and prizes, the winners of which were as follows—

First	(The Goodall Trophy and Replica)		
		G. L. Thornton	90–20–70
Second	(Mr. S. R. Meadows' prize)		
		J. E. Broadhurst	81–10–70
Third	(Mr. Fred Smith's prize)		
		J. S. Loxham	84–11–73
Scratch	(Mr. J. G. Hopkinson's prize)		
		P. W. Smith	79

The Society, and especially the competitors, are very greatly indebted to Mr. W. R. Leigh for his keenness and enthusiasm in organising both this meeting and the first annual competition, held at St. Annes-on-Sea in September 1953. Thanks are due also to the donors of the prizes.

Alfred William Carpenter
Presentation of the Society's Silver Medal

The Silver Medal of the Society of Dyers and Colourists was presented to Mr. A. W. Carpenter by Mr. F. L. Goodall (*President of the Society*) at the Annual Dinner of the Midlands Section held at the King's Head Hotel, Loughborough, Leicestershire, on Friday, 26th March 1954, presided over by Mr. S. R. Meadows. The President spoke of Mr. Carpenter's hard work and efficiency as Honorary Secretary of the Midlands Section since 1947, of the success of the Summer School held at Loughborough in 1952, and of the part he played in the entertainment following such dinners.

In reply, Mr. Carpenter said that he was deeply sensible of the honour the Society had conferred upon him, and his pleasure at receiving the medal at the hands of so worthy a man as our President was enhanced by the presence that evening of so many prominent officers of the Society. The award was conferred for his work as Honorary Secretary of the Midlands Section, and as such, reflected particular credit on the many chairmen and committee members who had so worthily aided his efforts. He regarded the Society as a very important influence in the world of science and technology; to work for it was an honour in itself. Such work may have been arduous, but it had been made very pleasant by the number of personal friendships he had formed. On account of business calls, his visits to Bradford and to other Sections were few and far between; he was therefore particularly pleased to have received so many letters of congratulation from members of other Sections. That the Midlands Section was now in a flourishing condition was due to the efforts of many loyal members; his own work had been built upon a very solid foundation laid by such men as Edwards and Schardt over the past thirty-five years. Those early days must have been heart-breaking to them, but they won through. He hoped to continue to serve the Society for many more years.

Marjorie Forbes
Presentation of the Society's Bronze Medal

The Bronze Medal of the Society of Dyers and Colourists was presented to Miss M. Forbes by Mr. Fred Smith (*President-elect of the Society*) at a meeting of the West Riding Section held at the Victoria Hotel, Bradford, on 11th March 1954, Mr. H. Foster in the chair. Mr. Smith referred to Miss Forbes' successes in the City and Guilds examinations in the technology of dyeing, and to her services to the Society as Vice-chairman and subsequently Honorary Secretary of the Bradford Junior Branch. She had been capable and energetic, and the manner in which she had pleaded the case of the Junior Branch before the West Riding Section Committee had been brilliant.

In reply, Miss Forbes said that, as a very junior "senior" member, she was very deeply honoured by the award. She regarded it as a tribute to the efforts of all the Junior Members, in whose hands lay, to a large extent, the future of the Society. Miss Forbes thanked the chairman and members of the West Riding Section Committee for their kindness during her term of office.

Harry Rose Hadfield
Presentation of the Society's Bronze Medal

The Bronze Medal of the Society of Dyers and Colourists was presented to Mr. H. R. Hadfield by Mr. F. L. Goodall (*President of the Society*) at a meeting of the Manchester Section held at the Textile Institute, Manchester, on 2nd April 1954, Mr. G. S. J. White in the chair. The President spoke of Mr. Hadfield's valuable services to the Society as Honorary Secretary of the Manchester Junior Branch since 1949, and wished him well in the office of Honorary Secretary of the Manchester Section, which he was now taking up.

In reply, Mr. Hadfield expressed his appreciation of the award and the pleasure he had derived from his work for the Society. He thanked members of the Manchester Section Committee for their help during his period of office.

Reports of Sections for the Session 1953–1954

HUDDERSFIELD SECTION

Once again we have to report a successful session, and the average attendance during the session has been much higher than last year. The standard of lectures has been high, and, in accordance with our usual practice, we have tried to get talks of a practical nature.

As in past years, we have had two joint meetings with outside bodies. The one with the Huddersfield Textile Society was on *Orlon Acrylic Fibre*, and the one with the Royal Institute of Chemistry on *Photography as a Scientific Implement*. Both were very good lectures and we had a good attendance.

The outstanding lecture was in January, by F. Thies of Coesfeld, Germany, who also showed a good film illustrating how the machines were made and used. At this lecture, held in the Large Hall of the Technical College, we had the record attendance of 160.

As in previous years, we held our Annual Dinner at the Princess Café, and this year we had a record attendance at a very enjoyable evening.

The gratitude of the Section is due to the Chairman and members of the Committee for their work during the past session.

H. TURNER

Honorary Secretary, Huddersfield Section

LONDON SECTION

The 1953–1954 session was one of consolidation for the London Section in that no new features were introduced.

The second London Lecture was successful from every point of view. The President of the Society and about 300 others attended to hear Mr. Donald King, of the Victoria and Albert Museum, give an entertaining and able lecture on *The Development of Textile Printing in London and the Home Counties*. Also, by the kindness of the directors of the Swaisland Fabric Printing Co., a demonstration of hand-block printing was given. We feel this series of special lectures to be a valuable asset to the Society.

Five meetings were held in the rooms of the Royal Society, and these were well attended with an average of 43 members and guests. In addition, one lecture was held at Luton, and as usual was appreciated by the local members.

On the social side the Dinner and Dance held in the New Year was well supported. A dinner was also held following the Annual General Meeting of the Section.

Our thanks are due to Mr. G. F. Wood, who completed his two years' term as Chairman. He is succeeded by Mr. R. A. McFarlane, and the new Vice-chairman is Dr. H. W. Ellis, who thus relinquishes the position of Honorary Secretary, which he has held for about fifteen years. This period has been one of considerable development for the London Section, and the thanks of all members go to Dr. Ellis for his enthusiasm and zeal, which made the innovations so successful.

The next session is the Jubilee session of the London Section, and we look forward to further successes as a fitting celebration of the event.

H. W. Taylor

Honorary Secretary, London Section

MANCHESTER SECTION

Nine meetings have been held in Manchester this session, but no external lectures were organised. The attendances, particularly in the earlier half of the session, were lower than in recent years. An exception was the Ladies' Evening: this annual opportunity for an uninhibited discussion about the deficiencies of the textile and related industries fully retained its appeal. The one-day symposium on *Continuous Dyeing*, at which five papers were read and discussed, was strongly supported.

It is disappointing to record once again that the Junior Branch meetings were patronised mainly by senior members of the section, although the average attendance was over 50.

Section meetings have again been held at the Textile Institute headquarters, and we thank the Council of the Institute for permitting the use of their lecture room. The Junior Branch held their meetings in the Reynolds Hall, and the Manchester College of Technology are thanked for their co-operation. Two committee meetings were held at the Manchester College of Technology, and we thank Dr. J. M. Preston for providing accommodation.

The Knecht memorial prize was won for 1952–1953 by Mr. P. Brocklehurst, who obtained First Class Honours in the degree of B.Sc.Tech. in Applied Chemistry (Colouring Matters) in July 1953.

The Annual Dance was again held at the Grand Hotel, Manchester, on 25th February 1954. Over 200 members and their guests attended this very successful function.

Mr. G. S. J. White has completed his first year of office as Chairman, with Mr. J. Boulton as Vice-chairman. The Honorary Secretary, Mr. A. S. Fern, retired in April 1954, after the statutory five-year term of office, and was succeeded by Mr. H. R. Hadfield.

Since 18th April 1953, when there were 595 members and 27 junior members, the following changes have taken place—

	Position at 19.2.54
New members	+31
Transfers from other sections ...	+12
Resignations	−10
Transfers to other sections ...	−13
Deaths	− 6
Net increase	+14

The junior membership has increased by 7 to 34.

We regret to record the deaths of Mr. F. Allison, Mr. G. E. Holden, and Mr. E. Tunhill.

A. S. Fern

Honorary Secretary, Manchester Section

MIDLANDS SECTION

During the session under review, eleven meetings of the Midlands Section took place as follows—Leicester 3, Nottingham 3, Loughborough 2, Derby, Hinckley, and Nuneaton 1 each. The average attendance was 75. In certain of these meetings we enjoyed the co-operation of the local Textile Societies of Leicester, Nottingham, and Hinckley, while the Derby lecture, held jointly with the British Association of Chemists, was a big success. The level of all papers presented was very high. The majority had a full practical flavour, and discussions were long and animated. The social activity of the Section continues and excellent times were enjoyed at the Ladies' Dinner Dance and the Dinner. At the latter function we were honoured by the presence of the President, the Honorary Secretary of the Society, Honorary Member John Barritt, the Secretary of the Society, the Editor of the *Journal*, and the Chairman of the West Riding Section. The President took this opportunity of presenting the Silver Medal of the Society to the Honorary Secretary of the Midlands Section, an award which has given great pleasure to the Section as a whole.

In common with other sections, increase in membership has not been spectacular, the "balance sheet" showing a net increase of 5, viz. new members 15, resignations 8, transfers from other sections 9, transfers to other sections 10; we also regret to report the sudden and tragic death at an early age of Mr. Alan Bray.

It is regretted that illness prevented our Chairman, S. R. Meadows Esq., from taking up his duties during the first half of the session; we are

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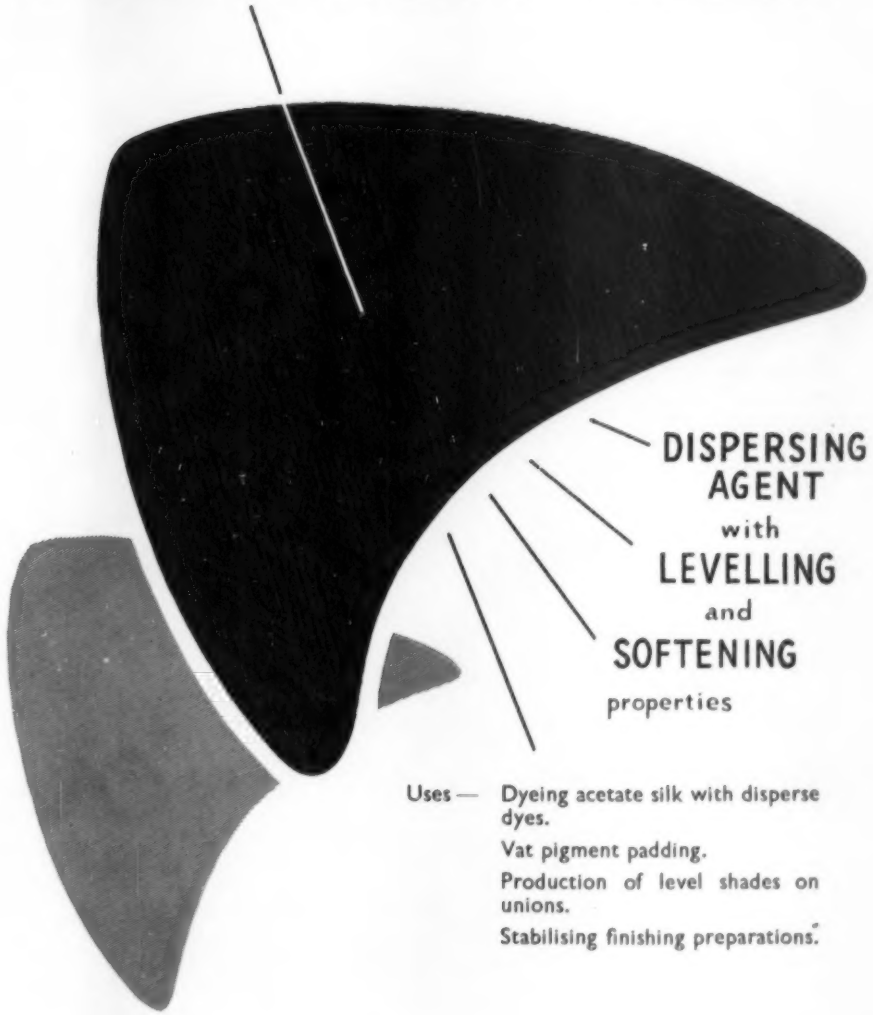

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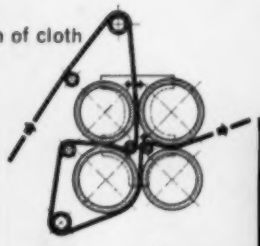

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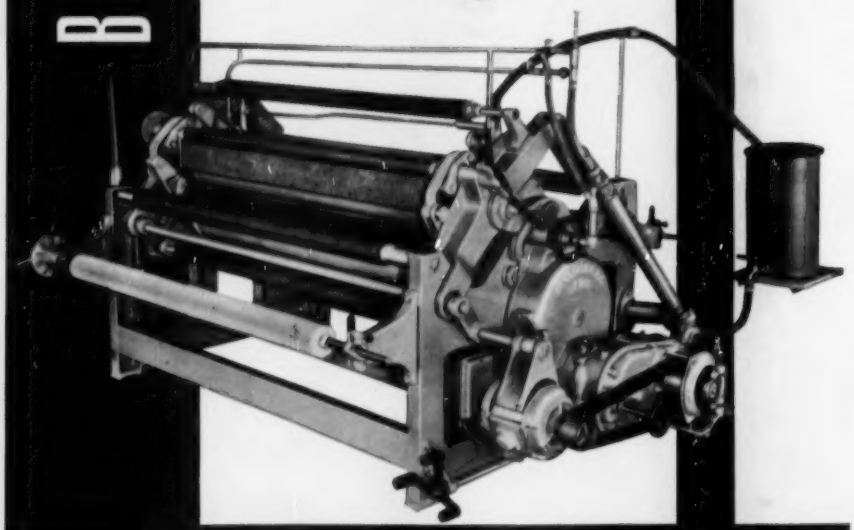

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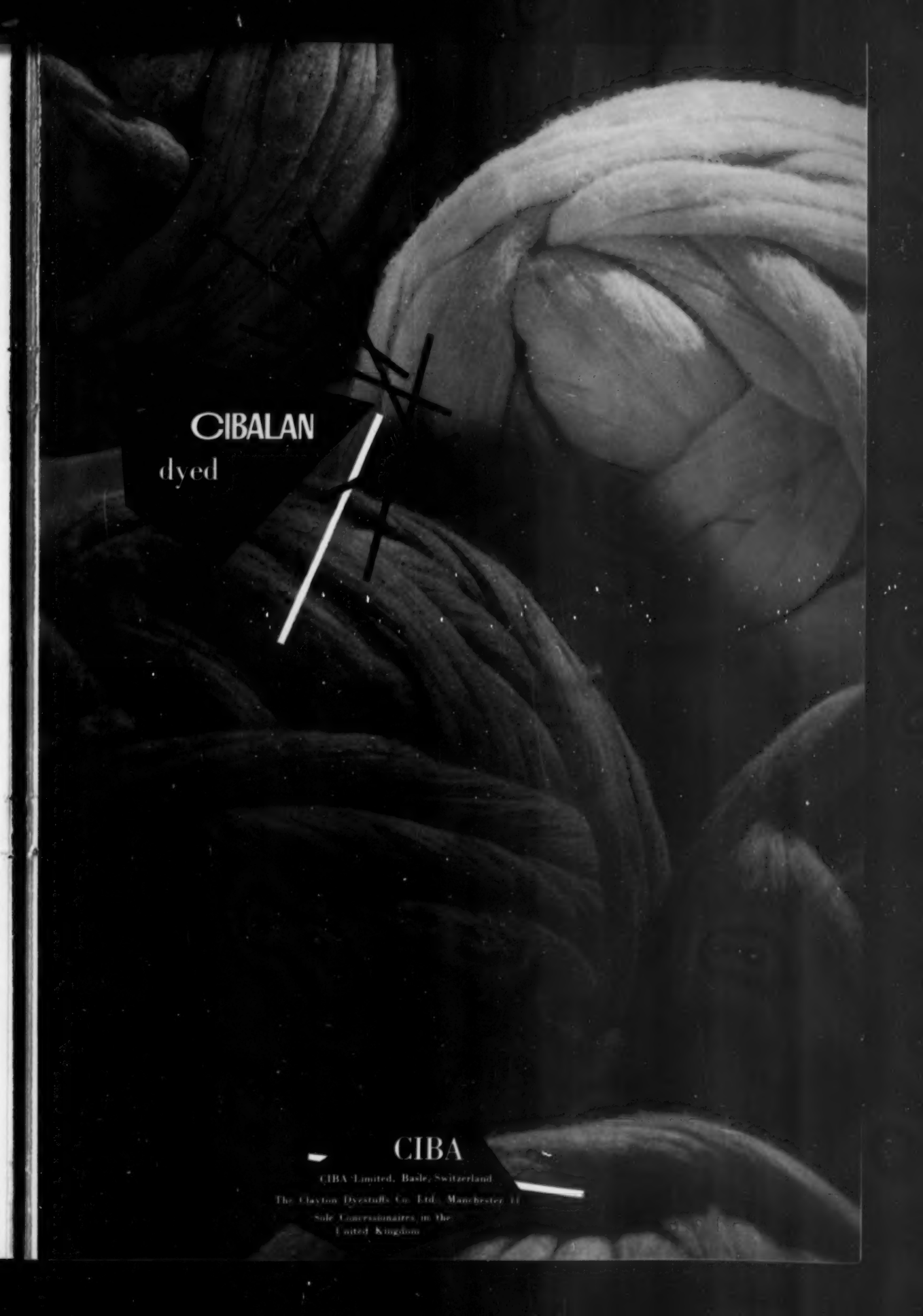
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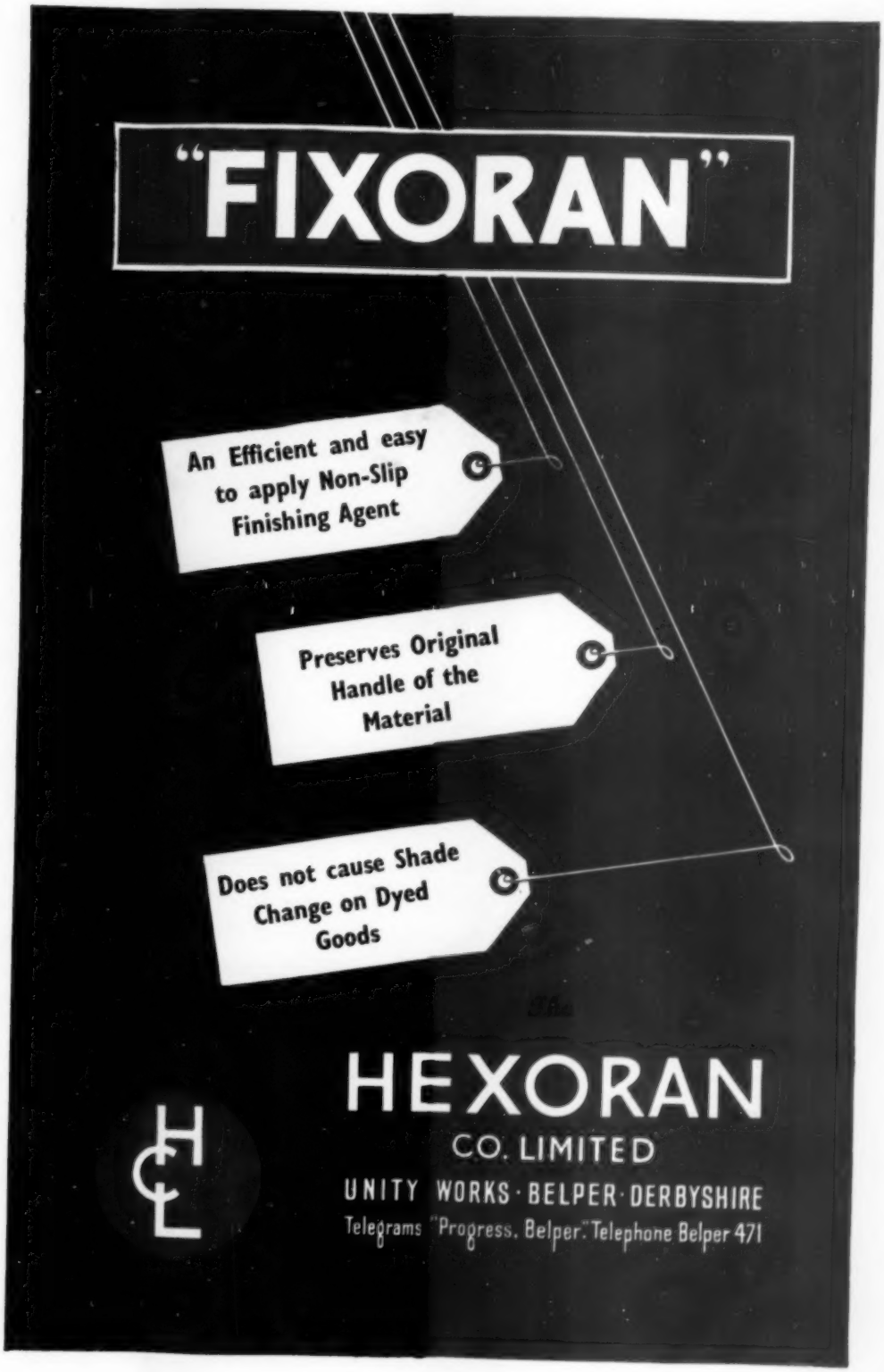
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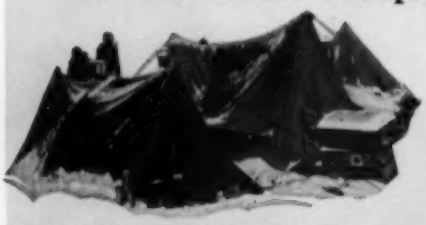

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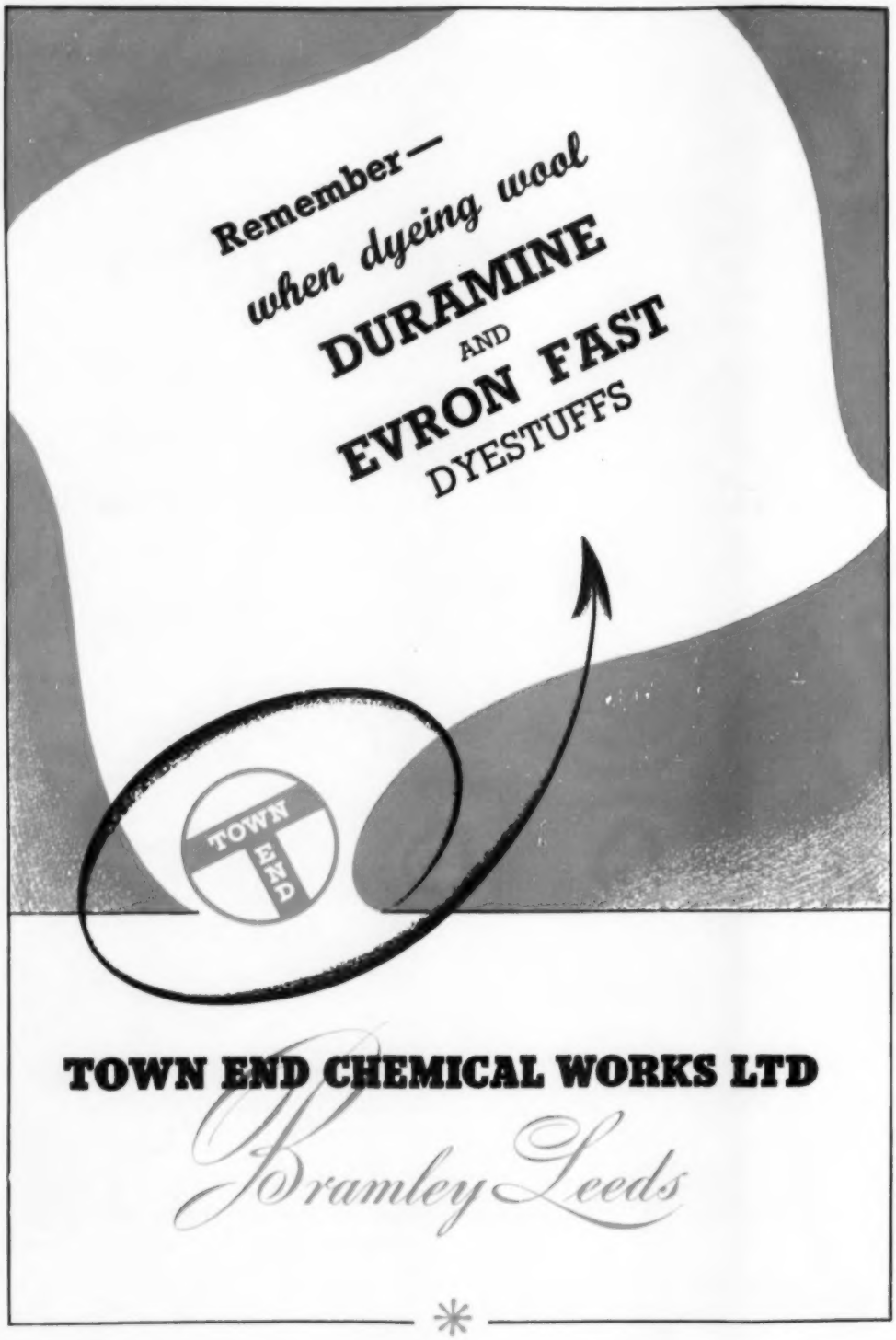
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pleased at his recovery. Mr. Meadows will continue in this post for a further twelve months, while Mr. C. A. Mills becomes Vice-chairman.

At the Annual General Meeting of the Section, the Chairman referred to the recent appeal by our President for honorary workers and expressed his gratification that Midlands members were taking a good share in spite of distance from headquarters. In addition, our Section had been well represented at meetings of Council. Mr. W. A. Edwards (Vice-president) and Mr. H. C. Olpin (Member of Council) had retired by rota, but we were happy to report the election to Council of Mr. A. G. Tyler.

Reference must be made to the retirement from the Midlands Section Committee of Mr. C. Schardt; in addition to having occupied the Chair, he has been a continuous and active member of our committee for no less than 31 years. Other committee members retiring by rota at our A.G.M. were J. V. A. Haywood, G. L. Thornton, R. Fernsby, and S. R. Meadows. Messrs Meadows and Fernsby were re-elected, while three newcomers are A. Datyner, G. Whitehead, and J. C. Wright. The undersigned was unanimously elected Honorary Secretary for a further period of office.

We are pleased that Midlands representatives have played an active part in formulating the Society's Diploma Scheme, and congratulate those who are honoured in the first list of Fellows, viz. Messrs. D. Carter, T. F. Heyes, E. R. Trotman, W. A. Edwards, H. C. Olpin, W. Penn, and A. G. Tyler (the last four being members of the Diplomas Committee).

Our final effort at the end of the session was the staging of an exhibition "Scientific Aids to the Dyeing Industry". Once more we were honoured by the presence of the President. The exhibition was a great success both in number and quality of exhibits and in attendance.

A. W. Carpenter
Honorary Secretary, Midlands Section

NORTHERN IRELAND

Under the chairmanship of Mr. J. Montgomery, in his second year of office, we can report a further successful year's working.

The most pleasing feature of the 1953–1954 session was an increased attendance at lectures. It is also gratifying to report that, although the potential for members in Ireland is limited, there has been an increase in membership.

Two of our lecture meetings were joint functions with the local branch of the Textile Institute, and these were most successful. We believe, as the result of our experience, that sectional co-operation is to the advantage of both organisations.

During the session we also held a Ladies' Evening. This consisted of a modest display of linen and rayon fabrics and dresses; berets and leather in various stages of production; and textile films were shown. Buffet supper was provided, and short non-technical talks were given on the exhibits. This function was very much appreciated, particularly by the ladies.

Our Annual General Meeting and Dinner were held on 27th March 1954. We had as our chief guests to dinner the President and the Honorary Secretary of the Society (Messrs. F. L. Goodall and J. G. Hopkinson) and the Minister of Commerce for Northern Ireland, the Right Honourable Lord Glentoran.

I would take this opportunity to express my best thanks to the Chairman, members of committee, and ordinary members for their support and co-operation during the session.

J. Porter
Honorary Secretary, Northern Ireland Section

SCOTTISH SECTION

A very successful and active session has been concluded by the Section, a pleasing feature being the continued good attendance at lectures. Seven meetings were held in Glasgow and one in Galashiels.

Membership shows an increase of 12 as compared with last year, which can be considered very encouraging.

The Annual General Meeting of the Section was held on Tuesday, 23rd March 1954. Mr. R. D. Alexander retired from the Chairmanship after two years, and this opportunity is taken of expressing our appreciation of his work for the Section and of extending a hearty welcome to his successor, Mr. R. A. Peel. Mr. J. G. B. McCallum was elected Vice-chairman, and Mr. P. J. Gough to fill a Committee vacancy.

It is with regret that we report the death of two of our members, Mr. R. Ritchie and Mr. D. Morton. Mr. Ritchie had a long association with the Society and was Honorary Secretary of the Section for eighteen years until 1937.

F. Atack
Honorary Secretary, Scottish Section

WEST RIDING SECTION

Under the Chairmanship of Mr. H. Foster, the Section has had an active year.

The two social events— the Supper and Smoker and the Ladies' Evening— arranged by Mr. J. M. Goodall, Honorary Social Secretary, were both outstandingly successful.

Lecture activities were fewer during this session, nine lectures being presented, which included one by Dr. H. J. White, of Princeton, in April 1953. Six other meetings were held in Bradford and two in Leeds; an innovation this year was that the Leeds meetings were held on Thursday evenings as part of the fortnightly series, rather than on Tuesday evenings and additional to the Bradford meetings.

Average attendance at the Leeds meetings was 40.

With regard to the Bradford meetings, adverse weather interfered with two meetings. The attendance at the opening lecture of the Session, when Sir Ernest Goodale, C.B.E., M.C., spoke on *Ancient and Modern Textile Production in London and the Home Counties*, was particularly influenced by severe fog. The average attendance at the Bradford lectures during the winter Session was 58.

The last lecture evening of the session was of particular interest, as on this occasion Miss Marjorie Forbes was presented with the Bronze Medal of the Society, for valuable services to the Society during her periods of Office as Vice-chairman and Honorary Secretary of the Bradford Junior Branch.

The Dyers' and Finishers' Association again gave £10 10s. 0d. for any scheme relating to the Junior Members of the West Riding Section. This took the form of competition papers as in previous years. Only two entries were received, but both were of high standard, and were awarded a first and a second prize.

The Members of the Section were invited to lectures held by the Bradford Textile Society and the Yorkshire Section of the Textile Institute, and the Section invited the Bradford Textile Society, the Bradford Industrial Safety and Welfare Organisation, and the Yorkshire Section of the Textile Institute to meetings.

The Committee met on two occasions.

G. E. STYAN

Honorary Secretary, West Riding Section

Exhibition of Scientific Aids to the Dyeing Industry

This venture of the Midlands Section was held in the Great Hall of the Leicester College of Art and Technology on Friday and Saturday, 28th and 29th May 1954. Both in quantity and in quality of exhibits, the show proved very satisfactory, and an excellent attendance was rewarding to the organisers.

At the opening ceremony, Mr. S. R. Meadows (*Chairman of the Midlands Section*) welcomed our President and the sister societies represented. Present on the platform were Mr. H. Scott (Midlands Hosiery Dyers Federation, Milanese and Warp Knitting Dyers Federation, and Lace Dyers Federation), Dr. D. Starkie (Hosiery and Allied Trades Research Association), Mr. J. C. MacCallum (Lace Research Association), Mr. W. Dutton (Textile Institute), Mr. C. F. Ward (Royal Institute of Chemistry and Society of Chemical Industry), and Mr. L. Priestley (representing the Principal of the College of Technology); also the organising committee, Messrs. A. W. Carpenter, A. Datyner, H. C. Olpin, and A. G. Tyler. He particularly wished to stress the invaluable work of Mr. A. Datyner (Senior Lecturer in Dyeing at the College) as Exhibition Secretary. This had entailed a lot of hard work; indeed, without his "on-the-spot" assistance and his peculiar knowledge of what was desirable to ensure success, the exhibition would not have been possible. Mr. Meadows then invited the guest of honour, the President of the Society (F. L. Goodall, Esq.), to open the exhibition.

In a brief speech, our President stressed the importance of science to modern industry; he referred to the enterprise of the Midlands Section in staging the exhibition and expressed his thanks to all those who had contributed, particularly to the College for its ready co-operation. He then declared the exhibition open.

On behalf of the dyeing trade, Mr. H. Scott expressed his gratification at the work the Society was doing for the industry. The present exhibition was but one example of the voluntary service of research and exchange of information that had grown up with the Society. In the present instance, he hoped that the exhibition would have an encouraging response as some recompense to Mr. Datyner and the committee for their efforts.

Mr. L. Priestley, as Head of the School of Chemistry and Dyeing at the College, said he was always happy to take part in any joint enterprise such as this. He regarded it as one of the privileges of the College to serve industry in this way. Had his Principal not been prevented from attending, he had no doubt that he would have expressed similar sentiments.

Thirty-four firms and research organisations took part in the display; within the bounds of the title, there was very little missing. Without unnecessary duplication, over two hundred pieces of apparatus were on show, some for the first time. Twenty firms provided continuous demonstrations. The exhibition attracted visitors from a wide area, including several from the North and one or two from overseas. There were also organised parties from various Midland technical colleges.

In addition to a show of publications, fastness standards, etc., our Society also staged a display of items of historical interest.

Our thanks are due to the many instrument manufacturers and research organisations, whose willingness to take part made it possible to stage such a comprehensive display with very little expenditure.

S R M

Northern Ireland Section Annual Golf Outing

The Annual Golf Outing of the Northern Ireland Section was held at the Massereene Golf Club, Antrim, on Saturday, 29th May 1954, at which there was a good attendance of members and visitors.

In the members' section the first prize was won by Mr. D. Kerr, while Mr. R. J. Hanna was runner-up. The visitors' prize was won by Mr. W. Brownlee, the runner-up being Mr. J. Holland.

Prizes were won also by Messrs. W. J. Watts and F. Heap.

J P

Indian Imports of Russian Dyes

India imported 497,900 lb. of Russian dyes in 1953 compared with 39,100 lb. in 1952. They formed almost 4% of the 1953 imports of dyes, and consisted mostly of cheap "bread-and-butter" dyes.

C O C

Trelon "Copolyamide" Fibre

Trelon (see J.S.D.C., **70**, 25 (Jan. 1954)) is described in *Faserforschung und Textiltechnik* (**5**, 171–172 (April 1954)) as being produced by reaction of a mixture of caprolactam and an equimolecular mixture of an $\alpha\omega$-diamine and an $\alpha\omega$-dicarboxylic acid. In this product, therefore, the group NH·CO is neither constantly oriented along the chain nor oriented alternately in the two directions. The first type of polymer "Eftrelon" is now in large-scale production, and a further type "Wetrelon" is to be produced (they are presumably designed for the manufacture of filament and staple fibre respectively). The properties of Eftrelon are given in detail (*loc. cit.*).

A E S

OBITUARY NOTICES

Robinson Percy Foulds

R. P. Foulds was born at Colne in Lancashire in October 1890, and was educated at Ackworth School and Manchester University, where he studied chemistry. During the First World War he served in the Friends' Ambulance Unit both in England and in France.

In 1919 he joined the newly established Research Department of the Tootal Broadhurst Lee Co. Ltd., where he remained until his untimely death on 26th March 1954, at the age of 63.

Foulds was a Master of Science, a Fellow of the Royal Institute of Chemistry, and a Fellow of the Textile Institute.

He had a wide knowledge of all textile processes from spinning to testing, which formed a valuable background for his research work; his unremitting experimental attack on textile problems, carried out with characteristic enthusiasm and a high degree of ingenuity and originality, often achieved results which surprised those who favoured a more formal approach. One of the earliest processes on which he was engaged was the controlled parchmentising of cotton, and one of the latest was the production of non-felting wool by the simple and direct use of bleaching powder. His greatest work, however, was in connection with the famous crease-resisting invention.

Foulds leaves a widow, four daughters, and two grandchildren; he also leaves a very wide circle of friends, all of whom will miss his cheerful personality and infectious gaiety.

J. T. Marsh

Wilfred Holt Oxley

We regret to record the death at the age of 48 years of W. H. Oxley, well known to members of the Society as Senior Director of J. C. Oxley's Dyes & Chemicals Ltd., formerly Secretary of the Bradford Junior Section, son of the late John C. Oxley (who was founder of the firm and pioneer of the British dyemaking industry after the 1914 war).

He was an ardent supporter of the Society, and subscribed generously to its development fund.

He was educated at Bradford Grammar School and Sedbergh School and received his final technical training under Dr. L. L. Lloyd at Bradford Technical College, after which he joined his father in the above firm, where he rose to the position of Chairman.

He was a keen golfer and lover of all sport, and adjutant to the Cleckheaton Army Cadets during the 1939 war. In 1935 he married Miss Clarice Gill, and they had three sons; all four survive him.

His charming culture and manner were unsurpassable.

Fred Smith

Frederick Oxley

We regret to record the death at the age of 49 years of Frederick Oxley, who survived his brother, W. H. Oxley, by only eight days.

Frederick was senior in age by one year, and like his brother was educated at Bradford Grammar School, Sedbergh School, and Bradford Technical College, after which he joined his father's firm of J. C. Oxley's Dyes & Chemicals Ltd., where he became Director and was employed as Technical Representative and Departmental Manager. During his time he visited Australia and New Zealand, where he arranged the firm's agencies.

In the 1939 war he was transferred to Messrs. Sandoz Chemicals Ltd., who were on munitions, on a process he would never disclose.

He was a keen cricketer and golfer. He married Elsie Halliwell, of Minehead, and they had two daughters; all three survive him.

His interest in the Society was mainly in the hidden support he gave along with his brother to any appeal made to him.

A man, never unfriendly, with a charming personality.

Fred Smith

New Books and Publications

Reports on the Progress of Applied Chemistry

Volume XXXVII 1952

London: Society of Chemical Industry. [1953.] Pp. 983. Price, 40s. 0d. (20s. 0d. S.C.I. members).

The annual review on the progress of applied chemistry grows yearly in size and matter. As before, it contains sections on— chemical engineering and water, fuel and fuel products, food and agriculture, inorganic chemistry, organic chemistry — biological products and textiles, plastics, adhesives, and paints. May we suggest that in future editions the chapters be numbered. Obviously, the reviewer must confine his observations to subject matters related to the dye and textile industries. R. K. Fourness and E. Stead report (pp. 493–523) that in 1952 the number of patents on "Intermediates and Dyestuffs" has increased. The authors give some statistics (p. 494) on dye production in various countries, amongst which no figures are stated for the United Kingdom, and for the U.S.A. the production figures for 1951 only and not for 1952; for other countries (Germany, Italy, Japan) the production figures for 1952 only go until June–July and lack comparison. In Table II export figures for the whole of 1951 are compared with the data for January–September 1952 (i.e. nine months only). Such statistics ought to be complete, or otherwise omitted altogether. The authors review in detail the progress in azo dyes and intermediates and with the anthraquinone, phthalocyanine, and miscellaneous dyes and brightening agents.

F. Courtney Harwood and R. E. Wagg report in their review on "Laundry Chemicals" (pp. 593–604) that progress has been well maintained during the period under review. As before, they have divided their report into subsections on— detergency, detergents, evaluation and analysis of detergents, and effects of builders (phosphates) and of cellulose derivatives when used with various detergents; they end their chapter with a review of brightening agents and dry-cleaning chemicals. Some of the literature quoted here is mentioned again in the report on "Detergents" (pp. 634–642) by L. V. Cocks, but this author reviews more fully the recent literature on the physical and technical properties of synthetic detergents, the raw materials, and manufacturing processes and the recent literature on analytical testing methods for synthetic detergents.

The chapter on "Cellulose Textile Chemistry" (pp. 723–743) does not catalogue the papers published in 1952, as this has been done in the *Review of Textile Progress* (published jointly by the Textile Institute and our Society), but the authors present articles on some special topics— J. Kenner on "Some Aspects of the Chemistry of Cellulose", D. W. Jones on "The Atomic Arrangement in the Ordered Regions of Cellulose", and A. Sharples on "The Form of Cellulose Derivatives in Solution and the Structure of Cellulose in the Solid State". All three authors are from the British Rayon Research Association. In his report on progress in "Non-cellulose Textile Chemistry" (pp. 744–763) F. O. Howitt includes a general review of the literature on wool research, shrinking and felting, scouring, bleaching, dyeing and finishing of wool; he quotes papers published on regenerated protein fibres and on synthetic fibres, on the dyeing of wool–cotton, wool–rayon and wool–nylon mixtures and on related subjects.

There are innumerable references to the literature in the book together with an author and subject index. The whole report is full of most useful information, and records great activity and progress in all sections of research and industry.

A. F. Kertess

The Standard Handbook of Textiles

By A. J. Hall, 4th edition 1954. Pp. viii + 306 with 209 Fig. London: The National Trade Press Ltd. Price, cloth, 25s. 0d.

This book was first published in 1946, a second edition appeared in 1947, a third and revised edition in 1950, and now a fourth, revised and enlarged edition. The first three editions have already been reviewed in the *Journal*— **62**, 387 (1946); **64**, 191 (1948); and **66**, 440 (1950).

This book contains much interesting information and gives a general picture of textiles with some 150 pages devoted to Bleaching, Dyeing, Printing, and Finishing, which probably constitute the best part of the book.

The minor errors printed in the first edition have been corrected; there has been a useful pruning of certain figures, and interesting new ones have been introduced.

Newer developments have been included, and those of most interest to readers of this *Journal* are fluorescent whitening agents, continuous bleaching and dyeing processes, and high temperature dyeing.

J. Barritt

An Introduction to Electronic Absorption Spectroscopy in Organic Chemistry

By A. E. Gillam and E. S. Stern. Pp. vii + 283. London: Edward Arnold (Publishers) Ltd., 1954. Price, 40s. 0d.

This timely, well written book is an excellent introduction to the subject of absorption spectroscopy as a tool to aid the organic chemist. As Prof. E. R. H. Jones says in the Foreword, it should prove to be most helpful to both undergraduate and postgraduate students. The first part is concerned with a description of the origin and basic laws of light absorption, the nomenclature employed, and the instruments and methods used for the determination and presentation of absorption spectra. A profusion of tables and absorption curves give data on the spectral characteristics of substances ranging from simple unsaturated compounds to complex natural products. Spectrophotometric methods used for the detection, identification, and analysis of organic compounds are described in detail, and examples are given

of the use of this tool in problems involving tautomerism and isomerism. Full literature references are given.

The scope of the book is limited to absorption in the ultraviolet and visible regions from 2,000 to 10,000 A., the greater part of the book being concerned with the ultraviolet. Apart from the special interests of the authors, it is perhaps natural that this should be so, since absorption in this region is shown by all unsaturated compounds whether coloured or colourless and, in questions of structure determination, gives more information. Nevertheless, it is felt that the book would profit greatly by an expansion of the sections on the visible absorption of dyes. The interpretation of their spectra, as a result of work by many chemists, particularly Brooker, may be said to have contributed as much as has the study of ultraviolet spectra to the establishment of empirical rules of absorption and to a better understanding of the relationship between resonance and light absorption.

The book is clearly printed, and the only errors noted were obvious ones not likely to mislead.

E. B. KNOTT

Methoden der Organischen Chemie (Houben–Weyl)

Band VII Teil 1

Sauerstoff-Verbindungen II

Aldehyde

By O. Bayer, edited by Eugen Müller. 4th edition 1954. Pp. xxii + 556. Stuttgart: Georg Thieme Verlag. Price, DM 82.

This volume is devoted entirely to the chemistry of aldehydes, and is prefaced by a general introduction to the nature and significance of the aldehyde group (–CHO) itself. The subject-matter is comprehensively treated in three main divisions—

A. Preparation of Aldehydes—This includes references to every method extant for the direct introduction of CHO, and for substitution of radicals containing aldehyde or acetal groups, for the oxidation of the methyl, primary alcoholic, etheric, aminomethyl, and chloromethyl groups to CHO, for the conversion of compounds at the same oxidation stage by transformation reactions, or by cleavage of heterocyclics, or from aliphatic nitro compounds, and for the reduction of carboxylic acids and of their functional derivatives; also by degradation processes, and from other aldehydes by reactions which preserve the aldehyde group intact.

B. Reactions of Aldehydes—These include transformations having special reference to aldehyde regeneration, and to the replacement and also removal of CHO groups.

C. Supplementary References to Aldehyde Preparation—These concern the reactivity of aldehydes, their stability towards oxidation, the stabilisation of aldehydes and acetals, purification, optically active aldehydes, and a brief mention of isotopic aldehydes containing ^{14}C and deuterium.

In all cases, the patent as well as the scientific literature has been thoroughly searched, many of the processes described have been experimentally confirmed and frequently improved, while many hitherto unpublished observations from the I.G. laboratories are included.

This volume, with its author index of 30 three-column pages and subject index of 22 two-column pages, will be welcomed by all who are interested in the subject.

H. H. HODGSON

Methoden der Organischen Chemie (Houben–Weyl)

Band II

Analytische Methoden

Edited by Eugen Müller. 4th edition 1953. Pp. xxiii + 1070. Stuttgart: Georg Thieme Verlag. Price, DM 139.

This compendium is self-contained, and includes all of the most important of the published analytical methods in organic chemistry. The descriptions of them are devised to enable the analyst to carry out any particular determination from the data recorded in the text, thereby saving the time which otherwise would have to be spent in consulting the original reference. The literature has been searched up to 1952. Some very special methods, however, such as the determination of methanol in ethanol, have not been included in this collection but appear in the appropriate preparative volume.

The book is divided into two main parts—Part I has introductory sections devoted to the qualitative detection and quantitative estimation of all the elements which occur in organic compounds. Following come sections devoted to the analytical determination of the various functional groups, which are dealt with in the sequence of hydrocarbon (e.g. alkyl), and those containing oxygen (e.g. hydroxyl, carbonyl, etc.), sulphur (e.g. thiols, sulphonic acids, etc.), and nitrogen (e.g. nitroso, nitro, amino, etc.). Macro, semimicro, and micro methods are given in all cases where published.

Part II includes gas analysis, methods for the determination of melting, freezing, boiling and condensation points, thermal analysis and the estimation of molecular organic compounds; chromatographic analysis, and the qualitative and quantitative estimation of solvent mixtures.

Some idea of the scope of this volume may be gained from the fact that the author index involves 32 pages of names in three columns, and the subject index 51 double-column pages.

The appreciation already recorded with respect to Volume VIII (cf. J.S.D.C., **69**, 133 (1953)) applies with equal force to its present companion.

H. H. HODGSON

Annual Reports on the Progress of Chemistry for 1952

Volume XLIX

Published by the Chemical Society. Pp. vi + 423. London, 1953. Price, 30s. 0d.

This authoritative summary of progress in fundamental chemistry covers six chapters and contains 3,902 references, of which many are composite.

Section I on *General and Physical Chemistry* is divided into four subsections, which deal with bond interactions, surface chemistry, electrolytes, and the kinetics of homogeneous reactions respectively. It is to be noted that the calculations of bond interactions for ethane by Hansen and Dennison are valuable, because they provide a complete treatment of a relatively large molecule. In surface phenomena the solid–liquid interface is the main theme, since this has not been reported on previously. Strong electrolytes, incomplete dissociation in salt solutions, acids, bases, and redox systems, are the topics reviewed under electrolytes. No substantial change in the theory of rate processes has been reported, but a widespread interest continues to centre upon radiation effects induced in aqueous systems, and there is an increasing feeling that our conceptions regarding the nature of the primary reacting entities and the particular form of the substrate with which they react, are too rudimentary. Despite the fact that almost every possible radical or ion has now been invoked to explain this or that aspect of radiation—chemical effects in water, together with almost every possible permutation of their formation and breakdown—results continue to appear which meet with no explanation on existing concepts, or which seem incompatible with those of other workers.

Section II on *Inorganic Chemistry* specially notes among 216 references, the considerable interest sustained in the study of chelate and other complex compounds formed between metal cations and a variety of ligands. Also, mention is made of the remarkable and unique new iron compound, di*cyclo*-pentadienyliron (ferrocene), the first compound to be prepared which contains only carbon, hydrogen, and iron; it is a stable orange diamagnetic compound which vaporises above 100°C. without decomposition into a monomeric undissociated vapour obeying the perfect-gas laws even at 400°C. Attention is likewise drawn to what is stated to be the first example of a coloured double hydride, viz. silver aluminium hydride $AgAlH_4$, which is obtained as a yellow–gold precipitate when ethereal solutions of lithium aluminium hydride and silver perchlorate are shaken together at −80°C. Further, the occurrence of the lanthanon elements as uranium-fission products is shown to have stimulated interest in the properties of the pure metals themselves.

Section III on *Organic Chemistry* not only covers work published during 1952, but a theoretical reaction is devoted to the more salient features of the chemistry of free radicals in solution, since that rapidly advancing field has not been reviewed since 1948. Also the mechanism of the ozonisation of aromatic compounds, last reported in 1947, now receives special attention in view of the wider application of recent theories to other contemporary studies of the reactivity of polynuclear aromatic systems. The most novel discoveries of the year are stated to be the recognition of di*cyclo*pentadienyliron as a new type of symmetrical structure showing typical aromatic behaviour, and the use of clathrate compounds for optical resolutions. In synthetic organic chemistry the year has been notable for a stereospecific total synthesis of cortisone, and syntheses of flavin-adenine-dinucleoxide, morphine, and of all-*trans*-methylbixin. Each of these considerable achievements exemplifies in some way or other the high degree of specificity of modern techniques in organic chemistry, to which has been added a welcome catalytic hydrogenation method, by a lead and quinoline-poisoned palladium catalyst, for reducing triple to double bonds. The structure of lanosterol has now been established, the stereochemistry of the β-amyrin and lupeol groups of triterpenes has been elucidated, and an antibiotic isolated which incorporates every known type of carbon–carbon unsaturation. The increasing use of enzymic methods is seen in recent work on nucleotides and macromolecules. It is also the turn of polysaccharides to receive the main emphasis in a year which, in addition to furnishing its full quota of studies on new and rare varieties, has seen some readjustments in widely accepted concepts of the structures of such well known substances as starch, glycogen, and dextran. Structural studies have been facilitated by the continued use of chromatographic techniques for the fractionation of oligosaccharides on filter paper, on charcoal columns, and, as their acetates, on columns of Silene EF. For the chemistry of proteins, attention is drawn to five reviews during 1952 respectively by K. Lorane in *Quarterly Reviews*, by Edsall and by Boman in *Nature*, of a Faraday Society discussion, and in a Symposium of the Second International Congress of Biochemistry in Paris.

Section IV on *Biochemistry* reviews bacterial nutrition, vitamins, phosphate metabolism, and the thyroid hormone.

Section V on *Analytical Chemistry* endeavours with 803 references to review a fair distribution of work during 1952 classified under reagents, inorganic qualitative, gravimetric, and titrimetric analysis, indicators and related topics, classical organic analysis, instrumental methods (electroanalysis, coulometry and related methods, polarography, amperometric and potentiometric titrations, conductance methods, colorimetry and absorptiometry, nephelometry, fluorimetry, emission spectrography, absorption spectra, and chemical microscopy), and physical separation methods (adsorption chromatography, ion exchange, extraction, partition chromatography, for which the individual publications probably outnumber those in any other comparable branch of analytical chemistry, and ionophoresis and electrophoresis).

Section VI on *Crystallography* deals with crystal growth, the technique of structure analysis, and structural chemistry over the whole inorganic and organic field.

Comprehensive author and subject indexes complete a volume which will be a boon to every brand of chemist.

H. H. Hodgson

Abstracts from British and Foreign Journals and Patents

The titles of abstracts may be modified. Abbreviations of names of firms are listed in J.S.D.C., **68**, *23 (Jan. 1952), and also, together with symbols and the periodicals abstracted, in the annual index*

I— PLANT; MACHINERY; BUILDINGS

Static Electricity and its Removal. H. F. Schwenkhagen. *Melliand Textilber.*, **34**, 1182–1185 (Dec. 1953).

The Dominit ioniser, made by Dominitwerke G.m.b.H. (Hoppecke, Kreis Brilon, Westphalia), consists of a spray discharge rod and transformer. It uses high tension of 6 kv. (dangerous), and is very efficient in removing static electricity in the experiments reported. H. E. N.

PATENTS

Treating One Substance with Another, One of them at least being a Liquid. Tootal Broadhurst Lee Co. *BP* 708,118

When treating a solid or liquid with a liquid, e.g. when dissolving urea in formalin or caustic soda in butanol, the liquid flows in a closed circuit in which it goes down a central column from which it is centrifuged and is then forced to ascend in a more or less annular stream whereupon it is constrained to flow inwardly and downwardly in a second annular stream and finally inwardly and downwardly to rejoin the central column. The process may also be used for treating textiles with liquids, e.g. socks may be placed in an annular cage through which the liquid passes in either its upward or downward annular passage. C. O. C.

Facilitating the Insertion and Location of Liquid Supply Troughs into Machines. Timsons. *BP* 708,272

Thread-advancing Reel having a Definite and Constant Let-off Point. Courtaulds. *BP* 706,539

Thread Advancing Reel. Courtaulds. *BP* 708,223

Wet Processing of a Tow of Continuous Filaments. Celanese Corpn. of America. *BP* 707,771

After-Treating Freshly Spun Rayon Threads. Aktiebolaget Svenskt Konstsilke. *BP* 707,419

In the aftertreatment, e.g. washing, of freshly spun viscose rayon travelling in the form of layers of convolutions on a conveyor, the thread is covered by a protecting cloth which advances with the conveyor and prevents displacement of the convolutions by the treating liquor. W. G. C.

Spraying Oil or Water on Textiles. R. Schlumberger. *BP* 708,520

An atomiser for oiling or moistening textiles, e.g. cotton lap, is so devised that the spray is automatically cut off if the flow of material stops, thus avoiding local saturation. The pressure in the system is fully maintained so that the spray operates again immediately the fabric starts to move. J. W. B.

Hank Dyeing Machine. Freeman, Taylor Machines. *BP* 707,874

The opposite sides and ends of the frame or cage supporting the hanks extend downwards below the points at which the hank poles are supported. These extensions and the opposing walls of the vat are so formed that when the frame is lowered into the vat they engage so as to form a water-type seal extending around the frame. This prevents short circuiting of the liquor between the frame and the walls of the vat. C. O. C.

Yarn Carrier and Adapter for Package Dyeing. Russell Manufacturing Co. *USP* 2,640,341

Supporting Brackets for Textile Batching Devices. ICI. *BP* 706,977

Automatic Control of the Torque Changer Transmitting Power to the Mandrel of a Roll on which Cloth is Wound or Unwound. Établissements Collins & Tournadre. *BP* 706,459

Continuous Open-width Dyeing Machine. Rodney Hunt Machine Co. *USP* 2,641,121

An enclosed tank has a ribbed or corrugated bottom. The trough-like depressions in the bottom receive rolls taking up their major portions so as to form passages for fabric led under the rolls. The fabric passes upwards in the tank and over idler rollers placed above and parallel to the convex crests of the ribs of the tank's bottom. The fabric is first led beneath one of the bottom rolls, then over an idler roll and downwards to beneath the next bottom roll and so on. This enables dyeing with maximum uniformity and speed with a minimum of dye liquor. C. O. C.

Conservation of Heat Energy in Heating, Drying and other Industrial Processes. W. W. Spooner. *BP* 706,472

A waste heat source, especially low pressure steam mixed with air, e.g. from a drying cylinder, is used to raise steam at low pressure, this pressure being then raised mechanically, e.g. by a pump or ejector. C. O. C.

Removal of Curl from Knitted Fabrics. Wolsey. *BP* 707,053

Curling at the edges of cut knitted fabric in its delivery to a stenter, calender, or roller-blowing machine is overcome by contact of the fabric edge with a rotating, flat metal disc extending into the nip of take-up rollers. The underface of the discs are roughened to provide an outward brushing action on the fabric. Discs operating at the opposite edges of a fabric may be adjusted to accommodate varying widths since individual flexible drives are provided. G. E. K.

Drying Machine for Warps, Cloth, etc. Specialities Development Corpn. *USP* 2,641,062

A machine intended for drying yarn which has been treated with a bonding agent, the yarn being kept under tension throughout. Breakage of the yarn and/or failure of the drying medium stops the drive and sets up immediate cooling of the drying chamber. C. O. C.

Festoon Drier. R. C. Parkes. *USP* 2,640,250

The poles carrying the loops can only rotate at predetermined places in the chamber. This prevents distortion of the loops. C. O. C.

Reducing Shrinkage in Tubular Knitted Fabrics. Redman Process International. *BP* 708,654

The fabric is passed lengthwise through a treatment zone in absence of longitudinal tension while internally expanding the fabric widthwise so as to replace the stitches into their original knitted form and restore the fabric to its normal condition. C. O. C.

Aligning Webs for Lamination. Shellmar Products Corpn. *USP* 2,641,416

Setting and Dyeing of Hosiery made from Nylon or other Synthetic Fibre. Bailey Dye Works. *USP* 2,641,120

An autoclave type of apparatus has several chambers through which the articles move continuously for setting, dyeing and drying in succession. Setting and drying are each effected in chambers filled with a hot vapour under pressure, e.g. high pressure steam, The whole apparatus is automatic. C. O. C.

Stretching Material Laterally while it is travelling longitudinally. B. F. Goodrich Co. *USP* 2,643,116

Cleaning Device for the Cylinders of Coating and Varnishing Machines. W. Mailander. *BP* 708,880

Hosiery Brand Marking. Milford-Astor. *BP* 707,469

Design or brand markings are applied to hosiery or other articles presented by a reciprocating platen to the die beneath which a pigmented foil is drawn. A reciprocating carriage following in the rear of the platen carries a fold of hosiery. When printing of the foot or welt is completed the carriage is withdrawn in advance of the platen which is thereby cleared for the next cycle. G. E. K.

Garment Presses. J. C. Binns and S. Newberry. *BP* 709,314

A press of high output, which occupies little floor space and needs only one operator. C. O. C.

Automatic Continuous Treatment of Hides by a Series of Processes. Tanners Research Corpn. *BP* 706,910

Milling or Emulsifying Paint and the like. R. A. Dolman. *BP* 706,996

Jet Mills for Finely Grinding Materials. Titan Co. *BP* 708,491

Machine for Developing Diazotype Prints by Heat. Kalle & Co. *BP* 707,918

Coating Textiles, Paper, Plastics, etc. with Aluminium by Vacuum Evaporation (X p. 323).

II— WATER AND EFFLUENTS

Case Histories of Stream Pollution. A.A.T.C.C. Committee on Stream Pollution. **I.** J. B. Dick; **II.** W. J. Snow; **III.** A. N. Henschel. *Amer. Dyestuff Rep.*, **43**, P112–P115 (15 Feb. 1954).

Three separate papers are presented in which details are given of actual plant construction and operation. J. W. B.

Effluent Treatment in the Viscose Rayon Industry. T. Kleinert and W. Wincor. *Melliand Textilber.*, **34**, 1166–1170 (Dec. 1953).

Evaporation of spinning baths and crystallisation of Glauber's salt involve losses in the effluent (per 1,000 kg. viscose staple) of 95–130 kg. SO_3 and 70–100 kg. NaOH. S lost in other ways amounts to 52–64 kg. (2 kg. as H_2S, 10–12 kg. as CS_2, 40–50 kg. as S and non-volatile cpd.). Flocking is greatly dependent on agitation of the effluent. The important factor appears to be not the type of agitation but that some agitation occurs when the effluent has aged to a certain extent. A dilute viscose soln. (0·30 g. cellulose per litre) in presence of 0·34 g. Na_2SO_4 per litre at 20°c. required at least 300 mg. free H_2SO_4 per litre for a rate of flocking sufficiently great for technical purposes. The same soln. in presence of 200 mg. free H_2SO_4 per litre flocked less rapidly as the concn. of Na_2SO_4 was increased from 0·34 to 40 g./litres but flocked more quickly again as the Na_2SO_4 concn. was increased even further. The time of flocking in presence of 200 mg. free H_2SO_4 per litre depended greatly on temp., being reduced by 80% as the temp. was raised from 10° to 25°c. and a further 15% on raising to 40°c. The ripeness of the xanthate has little effect. Sedimentation can be speeded by removal of small gas bubbles either by increasing or decreasing the pressure on the suspension, by stirring, or through aeration. The effects of temp., concn. of H_2SO_4 and Na_2SO_4, and of flock on the sediment volume were studied. Increased temp. has a beneficial effect at shorter times of sedimentation (2–3 hr.), but none at longer times (8 hr.). Increased concn. of H_2SO_4 slightly increases the sediment volume, of Na_2SO_4 decreases it, and of cellulose decreases it also. Flocking and sedimentation are best carried out on undiluted effluent, preferably with addition of aged sediment. H. E. N.

III— CHEMICALS; AUXILIARY PRODUCTS; FINISHING MATERIALS

Aliphatic Wool-wax Alcohols—A Review. H. W. Knol. *J. Amer. Oil Chem. Soc.*, **31**, 59–62 (Feb. 1954).

A review of the composition of wool–wax alcohols is given with references. P. G. M.

Laboratory Evaluation of Spinning Oils. A. Pariset. *Bull. Inst. Textile France*, **43**, 7–32 (Dec. 1953).

Laboratory methods for the evaluation of spinning oils, including storage tests on wool, are critically discussed. It is claimed that the tests described enable a quantitative assessment to be given to factors which have been known hitherto in only a qualitative manner. G. L.

Surface-active Properties of Salts of α-Sulphonated Acids and Esters. A. J. Stirton, J. K. Weil, and R. G. Bistline. *J. Amer. Oil Chem. Soc.*, **31**, 13–16 (Jan. 1954).

In a comparison of the solubility, detergency, and surface-active properties of the mono- and di-sodium salts of α-sulphonated lauric, myristic, palmitic, and stearic acids, it has been shown that the less soluble α-sulphopalmitates, α-sulphostearates, and sodium hydrogen α-sulphomyristate are the best detergents. The easily soluble disodium α-sulpholaurate resembles a simple electrolyte with little evidence of surface-active properties. Ammonium hydrogen α-sulphopalmitate and triethanolammonium hydrogen α-sulphopalmitate are more soluble surface-active agents and detergents than the sodium salts. Triethanolammonium hydrogen α-sulphopalmitate is exceedingly soluble in water at room temperature. The sodium salts of alkyl α-sulphopalmitates and α-sulphostearates are easily prepared from the isolated crude diacids. The esters of primary alcohols containing 1–6C are readily soluble in water and quite stable to hydrolysis, especially in acid solution. Salts of α-sulphonated esters of secondary alcohols are stable to acid and to alkaline hydrolysis. P. G. M.

Cellulose Carboxydextrins in Soap Boiling. L. Sergeev. *J. Appl. Chem. U.S.S.R.*, **27**, 352 (March 1954).

In a letter to the Editor the claims of Antŷkov (see J.S.D.C., **70**, 26 (Jan. 1954)) concerning the technical and economic value of cellulose carboxydextrins as detergent components are rejected. A. E. S.

Emulsion Polymers for the Textile Industry. L. Shapiro. *Amer. Dyestuff Rep.*, **43**, P132–P140 (1 March 1954).

Fundamental principles of emulsion polymerisation are reviewed, and the effects on emulsion properties (as applied to textiles) of variables such as copolymer composition and particle size, and film formation and emulsion plasticisation, are discussed. A list of textile applications covers the following— polyvinyl acetates, polystyrenes, vinylidene chloride copolymers, butadiene–styrene copolymers, acrylic ester copolymers, styrene–maleic copolymers, polyacrylic acid, and polyvinyl alcohols, the last three being water- or alkali-soluble. This is followed by a different classification, in which resins are listed in terms of their suitability for particular finishes, e.g. non-slip, water-repellant, upholstery backing, shrinkage- and crush-resistant, and warp sizing. J. W. B.

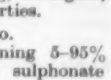
Structures of Basic Chromium Sulphate Complexes. S. G. Shuttleworth. *J. Soc. Leather Trades Chem.*, **38**, 58–71 (Feb. 1954).

Theories on basic chromium sulphate complexes are reviewed and criticised. J. W. B.

PATENTS

Emulsifying, Detergent and Wetting Agents. J. S. Vallernaud. *BP* 708,523

Oxazines of formula—



(R = acyl or acyloxy of >11C and connected to N by at least 2C; X^1 and X^2 each = 1 or 2 CH_2 groups) have in many cases emulsifying, detergent, wetting, bactericidal and insecticidal properties. C. O. C.

Detergents. Monsanto. *USP* 2,642,400

Compositions containing 5–95% of a sodium alkyl aromatic hydrocarbon sulphonate and 95–5% of an ethylene oxide–tertiary alkyl mercaptan condensate have much greater wetting and detergent properties than would be expected from adding together those of the individual components. C. O. C.

Latent Catalysts for Hardening Thermosetting Aminoplasts. N. Kingsley. *BP* 709,078

An alkyl ester of a *p*-alkylbenzenesulphonic acid (each alkyl of 1–4C) form neutral aqueous or aqueous-alcoholic solutions which are stable at room temperature but hydrolyse rapidly at 90–100°c. but not so rapidly that they cause polymerisation to be too vigorous with consequent formation of too brittle a plastic. Thus ethyl *p*-toluene sulphonate (2 parts by wt.) was dissolved in industrial methylated spirits (4) and the solution stirred into methylol melamine (100) in water (400). Cotton cloth impregnated with 12% of this solution and heated for 10 min. at 95°c. became shrink- and crease-resistant. C. O. C.

Fire-retarding Coatings composed of a Nitrogen-Phosphorous Compound with an Aldehyde Resin. Monsanto. *USP* 2,642,405

A fire retarding composition of great stability consists of an aqueous solution or dispersion of an aminoplast

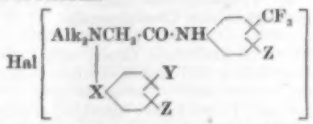
condensate and a compound containing N and P, e.g. a condensate of ammonia with phosphoryl chloride. C. O. C.

Basic Linear Polyureas and Polyurethanes. FBy. *BP* 706,717

High mol. wt. linear basic polyureas and polyurethanes are obtained by reacting in presence of a lower aliphatic alcohol a di*iso*cyanate or glycol-bis-chlorocarbonate with a diprimary aliphatic polyamine containing in the C chain at least one secondary amino group. The products may be used for rendering textile fibres water repellent or imparting to them affinity for acid dyes or they may be be used as binders in pigment printing. They can also be melt spun into fibres or cast as films. C. O. C.

Quaternary Ammonium Mothproofing Agents. Variapat. *BP* 706,421

Compounds of formula



(The 2 Alk together contain $<7C$; Z = Hal, H, or CF_3; Y = CF_3 or Hal; X = CH_2, $CH_2{\cdot}CONH$, $CH_2{\cdot}CH_2{\cdot}CO{\cdot}NH$, $CH_2{\cdot}CH_2O{\cdot}CO{\cdot}NH$, $CH_2{\cdot}CH_2{\cdot}NH{\cdot}SO_2$, $CH_2{\cdot}CH_2{\cdot}CH_2{\cdot}NH{\cdot}CO$, $CH_2{\cdot}CO{\cdot}O$, or $CH_2{\cdot}CH_2{\cdot}NH{\cdot}CO{\cdot}NH$) are very effective water-soluble mothproofing agents. C. O. C.

Solvents for Polyacrylonitrile. Eastman Kodak Co. *USP* 2,642,406

Compounds of formula $[(CH_2)_2N]_3P{:}O$, $R{\cdot}PO[N(CH_3)_2]_2$, $[(CH_3)_2N]_2PO{\cdot}N(C_2H_5)_2$ or $CH_3O{\cdot}PO[N(CH_3)_2]_2$ ($R = C_nH_{2n+1}$ (n = 1–3) or C_nH_{2n-1} (n = 2 or 3)) are useful solvents for acrylonitrile polymers and copolymers with the exception that the compounds having the fourth formula above are solvents only for copolymers of acrylonitrile. C. O. C.

Dimethyl Phosphite as Low Boiling Solvent for Copolymers of Acrylonitrile (VI p. 317).
Levelling Agents for Wool Dyes containing No or only One Sulphonic Acid Group (VIII p. 320).
Rendering Polyvinyl Acetate More Resistant to Heat, Organic Solvents, Water and Mechanical Deformation (XIII p. 326).
Standardisation of Methods of Test for Auxiliary Products (XIV p. 327).

IV—RAW MATERIALS; INTERMEDIATES; COLOURING MATTERS

Surface Films of Phenolic Compounds. A. J. G. Allan and J. H. Schulman. *J.C.S.*, 1238–1241 (April 1954).

The Langmuir trough technique has been used to investigate the surface structure of di- and poly-phenols, using conc. $(NH_4)_2SO_4$ soln. to make the compounds form insol. monolayers. The diphenols form hairpin structures when the hydrocarbon chain joining the phenolic groups contains more than three methylene groups. An analogy exists between the m.p.s of the compounds and their monolayer stability. The polyphenols form gel films in which the structure of the molecule is such that one phenolic group is oriented underneath the array of the polyphenolic chain. H. H. H.

Oxidation of Phenols with Diacyl Peroxides—II (A Simple Synthesis of *o*- and *p*-Hydroxyphenylacetic Acid or *iso*Coumaranone). F. Wessely and E. Schinzel. *Sitzungsber. Öster. Akad. Wiss. IIb*, **162**, (8), 969–991 (1953).

The reactions of diacyl peroxides, and in particular of diacetyl peroxide, with *o*-, *m*-, and *p*-cresol, and also with 2:4-dimethylphenyl, in glacial acetic acid, are described. The formation of the phenolic products, viz. deriv. of catechol and quinol, is explained on a radical mechanism. A simple synthesis of *o*- and *p*-hydroxyphenylacetic acid (or the corresponding *iso*coumaranones) is included, and the re-arrangement investigated of methyl-substituted *o*- and *p*-quinol acetates in hot glacial acetic acid. H. H. H.

Some Reactions of Phthalimide. R. O. Atkinson. *J.C.S.*, 1329–1330 (April 1954).

Phthalimide reacts with formaldehyde and dimethylamine in acetic acid to form *N*-dimethylaminomethylphthalimide, which can take part in carbon alkylations in a similar manner to Mannich bases (cf. Brewster and Eliel, *Organic Reactions*, **8**, 99 (1953)) and may provide a useful method for the preparation of primary amines difficultly accessible by other means. H. H. H.

Reaction of Sulphonation. XXXI—Initial Ratios of Naphthalenedisulphonic Acids. A. A. Spryskov and B. I. Karavaev. *J. Gen. Chem. U.S.S.R.*, **23**, 1712–1716 (Oct. 1953).

The monosulphonation of naphthalene and of its monosulphonic acids is carried out at 57°c., at which temperature no appreciable hydrolysis of the products occurs, and the products are analysed quantitatively (see J.S.D.C., **69**, 71 (Feb.), 313 (Aug. 1953)). It is confirmed that the 1- and 2-monosulphonic acids are formed in the ratio of 85 : 15. The 1-sulphonic acid gives 75% of 1:5-, 10% of 1:6-, and 15% of 1:3- + 1:7-disulphonic acids. The 2-sulphonic acid gives 67% of 1:6-, 21% of 1:3- + 1:7-, 1% of 2:6-, and 11% of 2:7-disulphonic acids. From these results it follows that disulphonation of naphthalene under non-hydrolysing conditions would yield 63·7% of 1:5-, 18·5% of 1:6-, 16·3% of 1:3- + 1:7-, 0·2% of 2:6-, and 1·7% of 2:7-naphthalenedisulphonic acids. It follows also that the more readily hydrolysable isomer is always formed initially in the greater amount. A. E. S.

Homolytic Aromatic Substitution. V—Partial Rate Factors for the Phenylation of Diphenyl. J. I. G. Cadogan, D. H. Hey, and G. H. Williams. *J.C.S.*, 794–797 (March 1954).

The rate of phenylation of diphenyl relative to that of nitrobenzene by phenyl radicals (generated from benzoyl peroxide) is measured, and the rate of attack relative to benzene is calculated. The partial rate factors obtained are discussed with reference to the predictions of molecular orbital theory. H. H. H.

Nitration and Hydroxylation of Phenetole, Phenyl *n*-Propyl Ether, and Acetophenone by Pernitrous Acid. R. B. Heslop and P. L. Robinson. *J.C.S.*, 1271–1273 (April 1954).

Reaction of pernitrous acid (cf. Halfpenny and Robinson, *ibid.*, 928–939 (1952)) with the title compounds leads to nitration and hydroxylation, the nature of the side-chain influencing the susceptibility of the nucleus and the nature of the products. The attack is restricted to the benzene nucleus with formation of 6:2:1-$R.C_6H_3(NO_2)OH$ compounds, considerable amounts of dinitro compounds from the two ethers, and a high yield of *m*-nitro deriv. from acetophenone. The mechanism proposed is that homolytic fission of pernitrous acid; $HO{\cdot}O{\cdot}NO \rightarrow OH + NO_2$ occurs, the OH enters the nucleus, preferentially in the *o*- or *p*- position to form an aryl radical, which then reacts with NO_2 to give an unstable molecular species that breaks down with elimination of H_2O, HNO_2 or H. H. H. H.

Aromaticity in Seven-membered Ring Systems. A. W. Johnson. *J.C.S.*, 1331–1340 (April 1954).

This Tilden lecture for 1953 reviews the chemistry of the tropolone field and contains 97 references; in particular, the results of Cook and of Haworth in this country and of Nozoe in Japan are described. It is pointed out that the substitution reactions of the tropolones have followed mainly the trends already well established for benzene, except that the Friedel–Crafts substitutions, the Gattermann and the Kolbe reactions, and chloromethylation cannot be applied to the tropolone ring system, possibly because of the formation of stable intermediate tropolone complexes. The widespread use of the alkaline rearrangement of tropolones to derivatives of benzoic acid, which is the basis of the interpretation of the course of the substitution reactions, depends on the fact that substituent groups, even in the α-positions, retain their relative positions in the ring during the rearrangement, which is of the benzilic type. H. H. H.

Azulenes and Related Substances. IV—Preparation of *cis-cyclo*-Pentane-1:2-diacetic Acid. S. Dev. *J. Indian Chem. Soc.*, **30**, 815–819 (Dec. 1953)

Tropolones. VII— Orientation in Tropolone. J. W. Cook, J. D. Loudon, and D. K. V. Steel. *J.C.S.*, 530–535 (Feb. 1954).

Nitration of tropolone yields γ- and a small quantity of α-nitrotropolone, which reduce with sodium dithionite to α- and γ-aminotropolone respectively, whose diazo reactions are investigated and found to be normal in the case of the γ- compound. α-Aminotropolone, however, is found to be less suited for diazo reactions, since HNO_2 converts it largely to salicylic acid. Nitration of α-bromotropolone affords α-bromo-γ-nitrotropolone, α- and γ-carboxytropolones are prepared, the latter through dehydrogenation of a condensation product of pimelic and oxalic esters. The α-bromo is found to be replaced more readily than a β-bromo substituent in bromotropolones by *p*-thiocresol, in pyridine. H. H. H.

Kinetics and Mechanism of Diazotisation. V— Reaction of the Ammonium Ion with Nitrous Acid in Strong Hydrochloric Acid. VI— Reaction of the Glycinium Ion with Nitrous Acid in Hydrochloric Acid. H. Schmid and R. Pfeifer. *Sitzungsber. Oster. Akad. Wiss. IIb*, **162**, (8), 829—841, 842–852 (1953).

V— The rate law for the reaction of the ammonium ion with nitrous acid in strong hydrochloric acid solutions of concn. 1–8 moles/litre is found to be in agreement with the authors' time law of 1938—

$$\frac{d(N_2)}{dt} = \frac{k a_{NH_4^+} a_{Cl^-} [HNO_2]}{a_{H_2O}}$$

VI— Similarly, the reaction of the glycinium ion, under the same conditions as in V, gives data in accord with the same rate law. H. H. H.

Reactions with Radioactive Nitrogen (^{15}N). XII— Mechanism of Phenyl Azide Formation by the Diazo Reaction. K. Clusius and H. Hürzeler. *Helv. Chim. Acta*, **37**, 798–804 (April 1954).

By means of ^{15}N it is shown that the mechanism of phenyl azide formation through the interaction of suitably marked diazonium and azide ions does not follow the classical conception of the azide ion becoming attached to the phenyl residue and the diazo nitrogen being evolved as N_2, but involves the formation of pentazen intermediate products resulting in a two-way reaction—

$$[C_6H_5\overset{a}{\bar{N}}{=}\overset{b}{N}I]^+ + [\overset{e}{\bar{N}}{=}\overset{d}{N}{=}\overset{c}{\bar{N}}]^- \rightarrow \begin{cases} \xrightarrow{85\%} C_6H_5{-}\overset{a}{N}{=}\overset{b}{N}{=}\overset{c}{N} + \overset{d}{N}{=}\overset{e}{N} \\ \xrightarrow{15\%} C_6H_5{-}\overset{a}{N}{=}\overset{e}{N}{=}\overset{d}{N} + \overset{c}{N}{=}\overset{b}{N} \end{cases}$$

The distribution is influenced by temp., concn., and substituents in the benzene ring. H. H. H.

Preparations of Aromatic Aldehydes and Ketones from Diazonium Salts. W. F. Beech, *J.S.C.*, 1297–1302 (April 1954).

A new method for preparing aromatic aldehydes and alkyl aryl ketones is described, viz., reaction of diazonium salts with formaldoxime, acetaldoxime, propionaldoxime, and acetaldehyde semicarbazone severally, under carefully controlled conditions, to give arylated deriv. which can be hydrolysed to aromatic aldehydes or ketones. Thus *o*-nitrobenzaldehyde is obtained from *o*-nitroaniline in 33% yield. The method succeeds, however, only to a limited extent with difunctional compounds. H. H. H.

Reaction of Phenyl-lithium with Azobenzene. P. F. Holt and B. P. Hughes. *J.C.S.*, 764 (Feb. 1954).

When LiC_6H_5(I) and azobenzene (II) react in equimol. proportions in dry ether under N_2 for ~ 5 min., triphenylhydrazine is formed in 10% yield at 20°c. and 20% at 0°c., together with hydrazobenzene. When the proportions are 2 mol. of I to 1 mol. of II and the period is 40 hr., then hydrazobenzene and diphenyl result. H. H. H.

Dyes having Antipyrin Nuclei— III. O. F. Ginzburg. *J. Gen. Chem. U.S.S.R.*, **23**, 1677–1682 (Oct. 1953).

By condensation of 1-naphthaldehyde and substituted benzaldehydes (*o*-, *m*-, and *p*-nitro-, *p*-chloro-, and 4-hydroxy-3-methoxy-) with antipyrin, further members of the diantipyrylphenylmethane series of dyes are prepared, and the dissociation constants of the carbinols of some of these dyes are determined (cf. J.S.D.C., **66**, 68 (Jan. 1950); **68**, 359 (Sept. 1952); **70**, 198 (May 1954)). Introduction of *p*-nitro or *p*-chloro in the phenyl group reduces the dissociation const. of the carbinol, i.e. leads to increased hydrolysis of the dye. Hydrolysis of dyes of this series is about 10 times as great in 60% acetone as in water. A. E. S.

Triphenylmethene Dye Radicals. E. Weitz, L. Müller, and K. Dinges. *Chem. Ber.*, **85**, 878–885 (1952): *Chem. Abs.*, **47**, 9948 (10 Oct. 1953).

Solutions containing dye radicals were prepared in a specially designed apparatus, by treating the dye salt in anhyd. pyridine with Zn dust, in an atmosphere of argon and nitrogen. Various derivatives were obtained from these solutions, e.g. treating the orange solution from crystal violet iodide with NO gave bis [tris(*p*-dimethylaminophenyl)methyl]-hydroxylamine. R. K. F.

Reactions of Triphenylmethane Derivatives with Organomagnesium Compounds. O. F. Ginzburg. *J. Gen. Chem. U.S.S.R.*, **23**, 1682–1685 (Oct. 1953).

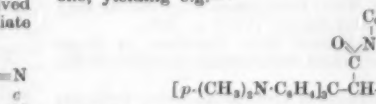
The carbinols of Malachite Green and Crystal Violet, and also corresponding derivatives of triphenylmethylamine and (triphenylmethyl)aniline, when treated with methylmagnesium iodide (Tserevitinov reaction) yield magnesium compounds that break down in soln., giving dye ions, e.g.—

$$[(CH_3)_2N{\cdot}C_6H_4]_3C{\cdot}O{\cdot}Mg{\cdot}X \rightarrow \{[(CH_3)_2N{\cdot}C_6H_4]_3C\}^+X^- + MgO$$

Further action of the Grignard reagent leads to formation of a deriv. of 1:1:1-triphenylethane. Diantipyrylphenylmethanol and 9:10-dihydro-9-hydroxy-10-methyl-9-phenylacridine react similarly. A. E. S.

Condensation Products of Triphenylmethanols with 3-Methyl-1-phenylpyrazol-5-one. I. O. F. Ginzburg and V. R. Terushkin. *J. Gen. Chem. U.S.S.R.*, **23**, 1049–1054 (June); **II.** O. F. Ginzburg. *Ibid.*, **23**, 1890–1892 (Nov. 1953).

9:10-Dihydro-9-hydroxy-10-methyl-9-phenylacridine and the carbinols of Malachite Green and Crystal Violet (and also triphenylmethanol itself under very vigorous conditions) condense with 3-methyl-1-phenylpyrazol-5-one, yielding e.g.—



$$[p\text{-}(CH_3)_2N{\cdot}C_6H_4]_3C{-}CH{-}C{\cdot}CH_3$$

The new C–C link is readily broken, e.g. by diazo compounds, which form the azo deriv. of the pyrazolone with regeneration of the carbinol. A. E. S.

Hydroxyfuchsone Dyes. XVIII— Halogen Derivatives of 4′-Hydroxy-3:3′-dimethoxyfuchsone. I. S. Ioffe and B. G. Belen'kii. *J. Gen. Chem. U.S.S.R.*, **23**, 1936–1939 (Nov. 1953).

The condensation of *o*-, *m*-, and *p*-chloro(and bromo) benzaldehydes with guaiacol proceeds similarly to that of *m*- and *p*-nitrobenzaldehydes, yielding the expected halogenated fuchsone dyes (see J.S.D.C., **70**, 198 (May 1954)). A. E. S.

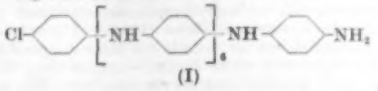
Ethylenic and Acetylenic Dyes. C. Dufraisse, A. Étienne, P. Barbieri, and B. Goffinet. *Compt. rend.*, **234**, 22–25 (1952): *Chem. Abs.*, **47**, 10231 (10 Oct. 1953).

A series of styrylcarbinol derivatives, e.g. (*p*-dimethylaminostyryl)diphenylcarbinol, were prepared and compared with the corresponding phenyl acetylenecarbinols. Both types give intense red dyes with acids. The acetylenic dyes regenerate the colourless alcohols with alkali, whereas the ethylenic dyes give mixtures of the original amino alcohol with one or more mesomeric forms. R. K. F.

Dyes obtained by Linear Condensation. I— Polymerisation of *p*-Chloroaniline and Allied Compounds. R. K. Kochar and S. Dutt. *J. Indian Chem. Soc.*, **30**, 829–835 (Dec. 1953).

Condensation of *p*-chloroaniline, *o*-chloroaniline, *p*-phenylene-diamine and *p*-bromo- or *p*-chloro-iodobenzene, *o*-phenylenediamine hydrochloride and *p*-dichloro- or *p*-chloroido-benzene, benzidine and *p*-chloro or *p*-bromo-iodobenzene, *o*-dianisidine and *p*- or *o*-dichlorobenzene or *p*-bromo- or *p*-chloro-iodobenzene, by heating with copper powder either dry or in nitrobenzene at 120°c. for 1 hr.

and at 180–200c. for 12 hr. in N_2 yields linear polymers containing 8 benzene or diphenyl residues (e.g. I from *p*-chloroaniline). These are leuco dyes, and are readily oxidised by air or O_2 to blue to violet–black dyes containing two double bonds ($TiCl_3$ determination) and thought to be analogous to Emeraldine. The dyes are readily soluble in dil. acetic acid, from which nylon is dyed in brilliant colours of good wet fastness.



(I)

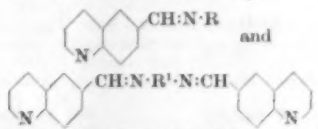
A. J.

Derivatives of Acetoacetic Acid. VI—A Synthesis of 1:3-Oxazine Derivatives employing Diketen. R. N. Lacey. *J.C.S.*, 845–849 (March 1954).

Diketen reacts with *NN'*-disubstituted deriv. of *S*-alkylthiourea to give 2-imino-1:3-oxazine deriv. with the elimination of thiol. The reaction is general, and the substituents may be alkyl, aryl, or aralkyl. Acid hydrolysis of the products gives dihydro-2:4-dioxo-1:3-oxazines, with elimination of amine, and, in certain cases, uracils, arising from the rearrangement of the imino-oxazine. The dihydro-dioxo-1:3-oxazines react with NH_3 and with alkyl- acid aralkyl-amines to give substituted uracils, affording a new route of wide applicability to these compounds. H. H. H.

Reactions of Quinoline-6-aldehyde. B. P. Lugovkin. *J. Gen. Chem. U.S.S.R.*, **23**, 1696–1703 (Oct. 1953).

Quinoline-6-aldehyde is now accessible, being obtainable from 6-methylquinoline by treatment with SeO_2 (Rodionov and Berkengeim, cf. *Chem. Abs.*, **39**, 4076 (1945)). Its general reactions are examined, and among derivatives prepared are a number of azamethin dyes, viz.—



and their methiodides (R = 2-pyridyl, 1- and 2-naphthyl; R^1 = *o*- and *p*-phenylene, *pp'*-diphenylene). A. E. S.

Preparation of Cyclic Ammonium Salt Dyes. V—Dyes of the Quaternary 2-Aminopyridine Salts. M. Katayanagi. *J. Pharm. Soc. Japan*, **68**, 228–231 (1948); **VI—Reaction of Anils with the Active Methyl Group 1.** *Ibid.*, 232–4; **VII—2.** *Ibid.*, 238–240: *Chem. Abs.*, **48**, 4545 (25 April 1954).

V— $HC(OC_2H_5)_3$ with 2-aminopyridine gave *NN'*-di-2-pyridylformamidine as colourless needles. Heating this product with cyclic ammonium salts containing an active CH_2 radical gave anilo-type dyes, e.g. heating it with lepidine-1-oxide methiodide yielded the red 4-[2-pyridylaminoethenyl]quinoline 1-oxide methiodide. The absorption maximum of the anilo-type dyes, especially those from 2-aminopyridine, had wavelength 400 A. greater than those of corresponding phenylamino compounds. Azomethin cyanine dyes on the other hand showed an absorption maximum 1500 A. less than those of the corresponding carbocyanine compounds.

VI— Anils, e.g. Schiff bases and $C_6H_5CH(N{:}CHC_6H_5)_2$, treated with cyclic ammonium salts having an active CH_2 radical yield styryl dyes of cyclic ammonium salts even in absence of piperidine as catalyst. In the same way, 8-methylbenzothiocarbocyanine is formed from anils having a CH_3 radical in 8-position, giving 8-phenylvinylcyanine but in this case piperidine is necessary as catalyst. This reaction can be used for cyclic ammonium salts in general and various Schiff bases.

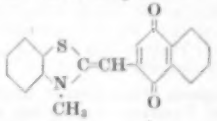
VII— In formation of styryl dyes from Schiff bases and cyclic ammonium salts containing an active CH_2 group, the effect of substituents in the aniline nucleus of the Schiff's base presence of polar radicals, e.g. NO_2, promotes the reaction. C. O. C.

Some Dyes derived from 2-Hydroxymethylbenzothiazole. R. A. Jeffreys. *J.C.S.*, 503—505 (Feb. 1954).

When 2-hydroxymethyl-3-methyl (I) and 3-methyl-2-methylthiobenzothiazolium toluene-*p*-sulphonate are refluxed together in ethanol with triethylamine (1 mol.), the orange dye bis-(3-methyl-2-benzothiazole)hydroxymethincyanine toluene-*p*-sulphonate is formed; its soln. in aq. ethanol is cerise, in pyridine purple, and in ethanol with a drop of triethylamine, blue, and all these colours slowly fade to become colourless. Similarly I and 2-2'-acetanilidovinyl-3-ethyl-benzoxazolium iodide give the corresponding trimethincyanine dye, which is orange–red in ethanol and turns to a transient green on addition of a drop of triethylamine. Two *merocyanine* dyes possessing a thiazolid-4-one ring are also prepared from the appropriate 5-ethoxymethylene intermediates, which are magenta in ethanol changing to blue on basification and then quickly fading to colourless. H. H. H.

Reactions of Methylene Bases of Nitrogen Heterocycles with Quinones. A. I. Kiprianov and A. V. Stetsenko. *J. Gen. Chem. U.S.S.R.*, **23**, 1912–1920 (Nov. 1953).

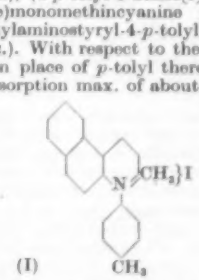
Methylene bases (containing $-NR{\cdot}C(:CH_2)-$, being formed by addition of alkali to a quaternary salt of an *a*-methyl-substituted nitrogen heterocycle) readily react with *o*- and *p*-quinones, yielding dyes of the *merocyanine* type. Absorption max. in neutral and acid soln. are listed for 13 dyes prepared by this method from bases of the benzothiazole, benzoselenazole, and quinoline series and various quinones. An example is—



A. E. S.

Cyanine Dyes. IV—Synthesis of 3-Methyl-4-*p*-tolylbenzo[f]quinolinium Iodide and Some of its Derivatives. G. T. Pilyugin. *Izvestiya Akad. Nauk S.S.S.R., Otdel. khim. nauk*, 1068–1073 (Nov.–Dec. 1953).

Acid condensation of paraldehyde with *N*-*p*-tolyl-2-naphthylamine yields the previously unknown 3-methyl-4-*p*-tolylbenzo[f]quinolinium iodide (I), which is converted by well known methods into the following dyes (wavelengths are those of absorption max. in alcoholic soln.)—bis(4-*p*-tolyl-3-benzo[f]quinoline)trimethincyanine iodide (644 mμ.), (4-*p*-tolyl-3-benzo[f]quinoline) (1:3:3-trimethyl-2-*pseudo*indole)trimethincyanine iodide (576 mμ.), (4-*p*-tolyl-3-benzo[f]quinoline) (3-ethyl-2-benzothiazole) trimethincyanine iodide (596 mμ.), (4-*p*-tolyl-3-benzo[f]-quinoline) (1-methyl-4-quinoline)monomethincyanine iodide (578 mμ.), (4-*p*-tolyl-3-benzo[f]quinoline) (3-ethyl-2-benzothiazole)monomethincyanine iodide (495 mμ.), and 3-*p*-dimethylaminostyryl-4-*p*-tolylbenzo[f]quinolinium iodide (543 mμ.). With respect to the corresponding dyes having ethyl in place of *p*-tolyl there is a bathochromic shift of the absorption max. of about 10 mμ.



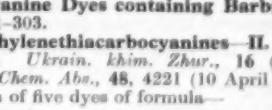
(I)

A. E. S.

Cyanine Dyes. IV—Syntheses of Aminovinyl Compounds and Carbocyanine Dyes from Imido and Cyano Compounds. H. Zenno. *J. Pharm. Soc. Japan*, **73**, 298–300 (1953); **V—Syntheses of *mero*-Carbocyanine Dyes containing Barbituric Acid.** *Ibid.*, 301–303.

αβ-Polymethylenethiacarbocyanines—II. I. K. Ushenko. *Ukrain. khim. Zhur.*, **16** (4), 450–453 (1950): *Chem. Abs.*, **48**, 4221 (10 April 1945).

Preparation of five dyes of formula—



(R' = C_2H_5 or C_6H_5; R^2 = CH_3 or C_2H_5; n = 1 or 2) is described. C. O. C.

Spatial Configuration, Light Absorption, and Sensitising Effect of Cyanine Dyes. L. G. S. Brooker, F. L. White, D. W. Heseltine, G. H. Keyes, S. G. Dent, and E. J. Van Lare. *J. Phot. Sci.*, **1**, 173–183 (1953): *Chem. Abs.*, **48**, 3823 (10 April 1954).

Photographic sensitisers can be classified according to the freedom of relative motion of the parts of the molecule as "loose", "compact", or "crowded". The first two of these categories comprise planar and the third non-planar molecules. Compactness is especially favourable for dye aggregation. Sheppard's concentration between planarity and strong sensitisation is confirmed and amplified to stress the marked loss of sensitisation which can accompany slight distortion from planarity. C. O. C.

Quenching of the Fluorescence of Anthracene—Transition from Strong to Weak Quenching. H. W. Melhuish and W. S. Metcalf. *J.C.S.*, 976–979 (March 1954).

Quenching of fluorescence in soln. occurs when quencher mol. and excited mol. occupy contiguous positions in a liquid. Strong quenchers are limited by the viscosity-controlled rate of formation of neighbouring pairs, while weak quenchers are limited by the concn. of such pairs and the efficiency of collisions within them. An equation is derived from this model which describes the observed behaviour of quenchers of intermediate strength. A suitable photometer is described for the precise measurements of relative fluorescence intensities. H. H. H.

Synthesis of Organic Fluorescent Compounds. I—Synthesis of Ureides containing a Benzene or Naphthalene Nucleus and the Fluorescence of these Compounds. R. Oda, Z. Yoshida, and H. Masuda. *J. Chem. Soc. Japan, Ind. Chem. Sect.*, **54**, 540–542 (1951); **II—Synthesis of Ureides containing a Biphenyl Nucleus and the Fluorescence of these Compounds.** *Ibid.*, 54–53: *Chem. Abs.*, **48**, 1982 (25 Feb. 1954).

I—The preparation of many derivatives of mono ureidobenzene and naphthalene is described. Examination of their fluorescence under ultraviolet radiation shows that the ureido group has less effect than the amino group.

II—Several ureides prepared from benzidine, tolidine, and 4:4′-di-*o*-anisidine yielded alcoholic solutions which were more or less fluorescent in ultraviolet radiation. The fluorescence was weaker than that of ureides containing the naphthalene nucleus. C. O. C.

Synthesis of Organic Fluorescent Compounds. VI—Sulphonation of Triaryliminazolinones. Z. Yoshida and R. Oda. *J. Chem. Soc. Japan, Ind. Chem. Sect.*, **54**, 732–733 (1951); **VII—Optical Bleaching Effect of Sulphonated Imidazolinone Derivatives.** *Ibid.*, 734–735: *Chem. Abs.*, **48**, 3353 (25 March 1954).

VI—Disulphonated products are obtained when triaryliminazolinones are treated with H_2SO_4 (d. 1·8). They have nearly the same fluorescent colour in aqueous solution as the corresponding monosulphonated compound; a 0·1% solution in water of the sulphonated compound has rather stronger fluorescence than a 0·1% solution in alcohol of the monosulphonated compound.

VII—Sulphonated 4-5-diphenyliminazolimine, 4:5-diphenylthioiminazolinone and 1:4:5-triphenylimidazolinone are useful fluorescent brightening agents for wool and silk. Sulphonated 4-phenyl-5-(*p*-dimethylaminophenyl)iminazolinone tints wool and silk yellow. Sulphonated 3:3′-dimethyl-4-(4:5-diphenyliminazolinon-1-yl)-4′-acetamidodiphenyl is a fluorescent brightening agent for cotton. Other sulphonated di- and tri-arylimidazolinones were not useful as fluorescent brightening agents. C. O. C.

Chemistry of the Pechmann Dyes. E. Klingsberg. *Chem. Rev.*, **54**, 59–77 (1954): *Chem. Abs.*, **48**, 4476 (25 April 1954).

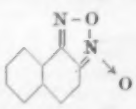
Review with 39 references. C. O. C.

Quinones. I—Directive Effects in the Substitution of Naphthaquinones. R. G. Cooke, H. Dowd, and W. Segal. *Australian J. Chem.*, **6**, 38–43 (1953).

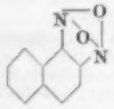
Properties of Hydroxynaphthaquinones. C. Kuroda. *Nat. Sci. Rept. Ochanomizo Univ.* (Tokyo), **2**, 87–92 (1951).

Naphthofuroxan and its Derivatives. I—Naphthofuroxan Bisulphite Compound. S. V. Bogdanov and B. I. Karavaev. *J. Gen. Chem. U.S.S.R.*, **23**, 1757—1761 (Oct. 1953); **II—1-Amino- and 1-Hydroxy-2-nitronaphthalene-4-sulphonic Acids.** S. V. Bogdanov and I. N. Koroleva. *Ibid.*, **23**, 1761–1764 (Oct. 1953).

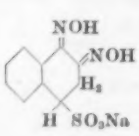
Naphthofuroxan, first prepared by Il'inskiĭ in 1886 by the oxidation of 1:2-naphthaquinone dioxime, was investigated by Green and Rowe (1913–1917), who at first considered it to have formula I, but later adopted formula II. Although some more recent workers have favoured formula I, the present authors consider that the question is still open and provisionally use formula II. They now, show that the bisulphite cpd. of 1:2-naphthaquinone dioxime III (readily obtained by the action of hydroxylamine in the cold on the bisulphite cpd. of 1-nitroso-2-naphthol) yields the corresponding naphthofuroxan deriv. IV on being oxidised with HNO_3, HNO_2, or H_2O_2. IV is readily reduced to 1:2-diaminonaphthalene-4-sulphonic acid. On treatment with Na_2CO_3 soln., IV yields at first 1-amino-2-nitronaphthalene-4-sulphonic acid (which can be obtained in 89% yield under suitable conditions) and other products; as treatment is prolonged at the boil, the primary products are converted into 1-hydroxy-2-nitronaphthalene-4-sulphonic acid (93% yield).



(I)



(II)



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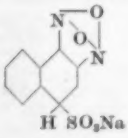


IV

A. E. S.

Chemistry of Anthraquinone Derivatives. W. Bradley. J.S.D.C., **70**, 57–63 (Feb. 1954).

The results are summarised of work on the chemistry of the anthraquinone vat dyes which has been carried out during the past five years in the Department of Colour Chemistry and Dyeing of the University of Leeds.

AUTHOR

Mobility of Halogen in 1-Halogenoanthraquinones—I and II. N. I. Artem'ev and A. E. Kretov. *J. Appl. Chem. U.S.S.R.*, **26**, 1276–1283, 1310–1313 (Dec. 1953).

I—The reactions occurring when a soln. of 1-chloroanthraquinone is heated in presence of weak alkalis and copper catalysts are studied. The nature of the copper catalyst (Cu, CuO, or CuCl) is of little importance, and the reactions are the same both in polar and in non-polar solvents, but are much more rapid in the former (e.g. nitrobenzene or diphenyl sulphone). When the alkali is an alkali-metal acetate or benzoate, the main product is anthraquinone, ~ 2% of 1-hydroxyanthraquinone and ~ 2% of 1:1′-dianthraquinonyl being formed as by-products; reaction is much more rapid with K salts than with Na salts. When an alkali-metal borate, carbonate, or silicate is used, the main reaction product is 1:1′-dianthraquinonyl.

II—Among the products of the reactions of 1-chloro- and 1-bromo-anthraquinone with alkali-metal acetate in presence of a copper catalyst are carbon dioxide and glycollic acid (isolated as the *p*-bromophenacyl ester). Reduction therefore occurs at the expense of the methyl group of the acetate. A. E. S.

4-Halogeno- and other Derivatives of Alizarin. B. S. Joshi, B. D. Tilak, and K. Venkataraman. *Proc. Indian Acad. Sci.*, **34A**, 304–315 (1951): *Chem. Abs.*, **47**, 8712 (10 Sept. 1953).

The preparations of 4-chloro-, 4-bromo and 4-iodo-alizarins and their conversion into a number of other derivatives are described. R. K. F.

Chromatographic Adsorption of Aminoanthraquinones on Alumina. N. R. Rao, K. H. Shah, and K. Venkataraman. *Proc. Indian Acad. Sci.*, **34A**, 355–367 (1951): *Chem. Abs.*, **47**, 8713 (10 Sept. 1953).

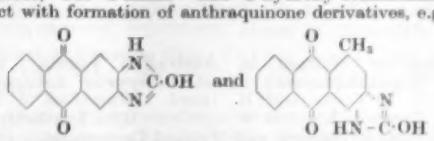
The strength of adsorption on Al_2O_3 from benzene solution, of a series of aminoanthraquinones increases in the following order— 1-methylamino-, 1-phenylamino-, 1:4-dimethylamino, 1-amino-, 1:5-diamino-, 2-methylamino-, 1:8-diamino-, 1-methylphenylamino-, 2-methylphenylamino-, 2-amino-, 1:4-diamino-, 1:4:5-triamino-, 1:2-diamino, 1:4:5:8-tetra-amino-, 2:6-diamino-. Explanations of these relative affinities in terms of chelation between NH_2 and CO, resonance between NH_2 and CO, the H bonding ability of amide groups and the electron-repulsing effect of CH_3 groups, are offered. R. K. F.

Islandicin (1:4:5-Trihydroxy-2-methylanthraquinone)— Synthesis from 3-Nitrophthalic Acid and *m*-Cresol. B. S. Joshi, B. D. Tilak, and K. Venkataraman. *Proc. Indian Acad. Sci.*, **32A**, 348–351 (1952): *Chem. Abs.*, **47**, 8712 (10 Sept. 1953)

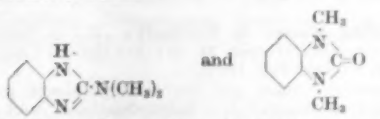
The constitution of islandicin was confirmed as 1:4:5-trihydroxy-2-methylanthraquinone by the following synthesis. 3-Nitrophthalic acid was condensed with *m*-cresol and the resulting product coupled with diazobenzene. On reducing with sodium hydrosulphite the azo compound thus obtained was reduced to 3-amino-2-(5-amino-2-hydroxy-*p*-tolyl)benzoic acid, the two NH_2 groups of which on diazotising and treating with H_2SO_4 being converted to OH groups; cyclisation occurring simultaneously. R. K. F.

Glyoxaline Derivatives. XI— Condensation of Phthalic Anhydride with Benziminazole Derivatives. L. S. Efros, B. A. Porai-Koshits, and S. G. Farbenshtein. *J. Gen. Chem. U.S.S.R.*, **23**, 1691–1696 (Oct. 1953).

Benziminazole and 2-methylbenziminazole do not condense with phthalic anhydride in presence of aluminium chloride, but 2-amino- and 2-hydroxy-benziminazoles react with formation of anthraquinone derivatives, e.g.—



from 2-hydroxy- and 2-hydroxy-5-methyl-benziminazole respectively. In order to determine which of the tautomers (e.g. –NH·C(OH):N– or –NH·CO·NH–) is active in this reaction, stable compounds having the two possible arrangements of bonds—



are submitted to the reaction. Both react very readily and it is concluded that the reactive species is the cation, e.g. [...NH·C(OH):NH...]$^+$, that may be formed by addition of a proton to either of the tautomers. A. E. S.

Phenanthraquinone Vat Dyes. R. D. Desai and N. S. Kumta. *J. Sci. and Ind. Research*, **11B**, 282–284 (1952): *Chem. Abs.*, **47**, 10230 (10 Oct. 1953).

A series of acyl and aroyl derivatives of 2-aminophenanthraquinone have been prepared and examined as vat dyes. Pale dyeings on cotton were obtained with the aroyl compounds, but in general and excepting the adipoyl derivative the *leuco* compounds have no affinity. A number of phenanthraquinonyl(anthraquinonyl)amines, prepared by condensing 2-bromophenanthraquinone and 3:6-dibromophenanthraquinone with 1-amino-, 2-amino-, or 1-amino-2-methylanthraquinone were found to be vat dyes of good fastness to light and soaping and moderately fast to bleaching. R. K. F.

Relation between Self-condensation and Substitution Processes in the Reactions of *meso*Benzanthrone with Anions. W. Bradley and F. K. Sutcliffe. *J.C.S.*, 708–711 (Feb. 1954).

Experiments with *meso*benzanthrone and a series of anions— KOH in CH_3OH, C_2H_5OH, or *iso*propanol, sodioaniline in aniline, and sodamide— show that anions which have the greatest tendency to form covalent links with carbonyl carbon (e.g., $C_6H_5^-$, NH_2^-) react by replacing nuclear hydrogen *ortho* or *para* to the CO group. Anions of higher electron affinity (e.g., OH^-, OR^-) function mainly as bases and cause the conversion of *meso*benzanthrone into 4:4′-dim*eso*benzanthronyl. H. H. H.

Benzanthrone and Dibenzanthrone Derivatives. P. N. Pandit, B. D. Tilak, and K. Venkataraman. *Proc. Indian Acad. Sci.*, **35A**, 159–165 (1952): *Chem. Abs.*, **47**, 8713 (10 Sept. 1953).

Debromination with Pd–$CaCO_3$ in presence of KOH and hydrazine in aq. CH_3OH, of 3-bromo-9-hydroxybenzanthrone, gave a mixture of 9-hydroxybenzanthrone and 3:12-dihydroxydibenzanthrone; and of 3-bromo-9-methoxybenzanthrone a mixture of the corresponding methoxy derivatives. The separation followed by reduction and diazotisation of 2-nitrobenzanthrone from the products of nitrating benzanthrone in acetic acid at 120°c. is also described. R. K. F.

Vat Dyes from the Condensation of Anthrone or Dianthronyl with Benzoquinone. T. Mine and S. Nakazawa. *J. Soc. Org. Synthet. Chem. Japan*, **11**, 227–229 (1953): *Chem. Abs.*, **47**, 10231 (10 Oct. 1953).

Comparison of the products obtained respectively by condensing anthrone with benzoquinone (modified procedure of *GP* 251,020) and by condensing dianthronyl with benzoquinone (modified procedure of *GP* 618,045) showed them to be identical, both giving purple vat dyes when reduced with sodium hydrosulphite. R. K. F.

Graphical Method for the Factorisation of the Secular Determinants of Aromatic Ring Systems on the Basis of the L.C.A.O.–M.O. Theory. E. Heilbronner. *Helv. Chim. Acta*, **37**, 913–921 (April 1954).

Molecular Orbitals in Homologous Series of Polynuclear Aromatic Hydrocarbons. I—Eigenvalues of L.C.A.O.–M.O.s in Homologous Series. E. Heilbronner. *Helv. Chim. Acta*, **37**, 921–935 (April 1954).

Chemistry of Coronene. III— Degradation to 1:12-Benzoperylene. R. Ott and A. Zinke. *Sitzungsber. Öster. Akad. Wiss., IIb*, **162** (9–10), 1132–1139 (1953).

Coronene is oxidised to *o*-coronenequinone by $Na_2Cr_2O_7$ in boiling glacial acetic acid–nitrobenzene solution, and the quinone degraded to 1:12-benzoperylene-6:7-dicarboxylic acid by aq. KOH and air, or H_2O_2, or Na_2O_2, in boiling dioxan. H. H. H.

4-Aminofluorene. W. J. P. Neish. *Rec. Trav. chim.*, **72**, 899–902 (Sept.–Oct. 1953).

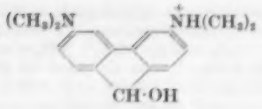
A synthesis of 4-aminofluorene from 4-nitrophenanthraquinone is described, in which the latter is reduced to 4-aminofluorenone, and thence converted by NH_2OH,HCl in pyridine to 4-aminofluorenone oxime, which is reduced to the title substance by 2·5% Na amalgam in acetic acid–ethanol. The ultraviolet-absorption spectra of the 2- and 4-aminofluorenes are recorded. H. H. H.

3:6-Disubstituted Fluorenes. II— Preparation of 3:6-Diaminofluorene from Fluorene, and the Attempted Internuclear Cyclisation of Derivatives of 4:4′-Diaminodiphenylmethane. A. Barker and C. C. Barker. *J.C.S.*, 870–873 (March 1954).

3:6-Diaminofluorene is prepared from fluorene in eight stages. Attempts at prep. of this amine, or of its deriv., from deriv. of 4:4′-diaminodiphenylmethane succeeded only with 4:4′-bisdimethylaminobenzophenone-2-diazonium sulphate, which afforded 3:6-bisdimethylaminofluorenone. H. H. H.

3:6-Disubstituted Fluorenes. III— Fluorene Analogues of Michler's Hydrol, Malachite Green, and Crystal Violet. A. Barker and C. C. Barker. *J.C.S.*, 1307–1309 (April 1954).

The preparation of the fluorene analogues in the title is described and their absorption spectra are recorded. The analogues of malachite green and crystal violet show a very large bathochromic shift when compared with the corresponding triphenylmethane dyes; the analogue of Michler's hydrol blue is colourless and has the structure (I).

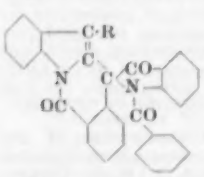


(I)

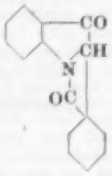
H. H. H.

Benzoyl Derivatives of Indigo— X. H. de Diesbach and M. Frossard. *Helv. Chim. Acta*, **37**, 701–705 (April 1954).

The formulæ of the Dessoulavy compound (A) and of Höchst Yellow R (B) were established in earlier papers, and the reactions of A and B with NH_3, pyridine, and aniline are now reported. A reacts with NH_3 to give *NN'*-dibenzoyldianthranilide and a red mixture from which an orange compound (C), a yellow compound (D), and a yellow substance of unknown constitution have been isolated. Decomp. of B by alkali gives E. Pyridine converts A into *NN'*-dibenzoyldianthranilide and Indigo Yellow 3G together with other indeterminate substances. The behaviour of A with aniline differs from that with other aromatic amines, and a mechanism for it is suggested.



(For A, R = Cl; for B, R = OH; for C, R = NH_2)



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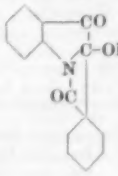
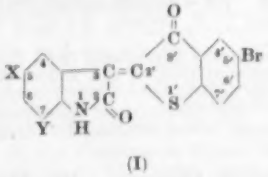


(E)

H. H. H.

Indigoid Vat Dyes of the Isatin Series. VII— 3-Indole-2′-(5′-bromo)thionaphthen-indigos. S. K. Guha and K. D. Banerjee. *J. Indian Chem. Soc.*, **30**, 820–822 (Dec. 1953).

3-Indole-, 3-(5-chloro)indole-, 3-(5-bromo)indole-, 3-(5:7-dibromo)indole-, 3-(5-bromo-7-nitro)indole-, and 3-(5:7-dinitro)indole-2′-(5′-bromo)thionaphthen-indigos have been prepared (I: X = H, Cl, Br, or NO_2; Y = H, Br, or NO_2). All are violet-red compounds, sol. in aniline, nitrobenzene, and tetralin, less so in alcohol, carbon tetrachloride, amyl alcohol, acetic acid, and acetone. The violet–red tetralin soln. becomes light yellow on continued boiling, reverting to the original colour on cooling for the first four compounds and to brownish yellow for the last two. 5-Chloro, 5-bromo, and 5:7-dibromo derivatives give light green soln. with conc. H_2SO_4, the others give blue soln. All are reduced in alkaline hydrosulphite to yellow vats which dye cotton. Compared with the corresponding 5′-methyl and 5′-chloro dyes, the colour intensity is 5-Br > 5-Cl > 5′-CH_3 (see Guha and Mallick, *ibid.*, **11**, 395 (1934) and **23**, 214 (1946)). Method of preparation is similar to that of 5′-CH_3 and 5′-Cl dyes (Guha and Mallick, *loc. cit.*).



(I)

A. J.

Derivatives of Acetoacetic Acid. VIII— Synthesis of Coumarins from Aryl Acetoacetates. R. N. Lacey. *J.C.S.*, 854–860 (March 1954).

Aryl acetoacetates, which are readily obtained by reaction of a phenol with diketen in the presence of a basic catalyst, e.g. triethylamine, are converted into coumarins by H_2SO_4. H. H. H.

Reactions of 4-Thionchromones with Amino Compounds, and with Methyl Iodide. W. Baker, G. G. Clarke, and J. B. Harborne. *J.C.S.*, 998–1002 (March 1954).

4-Thionchromones generally react with reagents containing primary amino groups to give deriv. of either chromone imine or *o*-hydroxyphenylpyrazole, although 2-methyl-4-thionchromone does not react with benzylamine. The most sensitive reagent for distinguishing between the two types of 4-thionchromone is hydrazine under mild conditions, which gives either a bright yellow chromone hydrazone or a colourless pyrazole; under vigorous conditions the product is in all cases the *o*-hydroxyphenylpyrazole. Methyl iodide reacts rapidly with 4-thionflavones to give methiodides which are hydrolysed by boiling water to flavones. H. H. H.

Survey of the Anthoxanthins. III— Paper Chromatography of Some Flavanones and Chalkones and their Glycosides— Isolation and Constitution of *iso*Butyrin, a Glycoside of the Flowers of *Butea frondosa*. B. Puri and T. R. Seshadri. *J. Sci. Ind. Research (India)*, **12B**, 462–466 (1953): *Chem. Abs.*, **48**, 4773 (25 April 1954).

Chalkones gave yellow or orange spots on paper chromatograms developed with C_6H_5OH–H_2O, the flavanones were colourless and were detected by spraying with NH_3 or $FeCl_3$. Naringin and hesperidin are wholly of flavanone structure even when hydrolysed to naringenin and hesperetin. However, butrin on hydrolysis yields a mixture of butein (chalkone) and butin (flavanone). Extraction of the flowers of *Butea frondosa* yielded a mixture of butrin and a new glycoside *iso*butyrin of lower R_f value. *iso*Butyrin was separated from butrin by fractional crystallisation from CH_3OH and was obtained as yellow crystals giving olive brown with alcoholic $FeCl_3$. Hydrolysis of *iso*butyrin with 7% H_2SO_4 gave 2 mol. of glucose; methylation followed by hydrolysis yielded 2:4′-dimethylbutein. Butrin was converted to *iso*butyrin by treatment with 5% NaOH. *iso*Butyrin is thus the 4:3′-diglycoside of butein. C. O. C.

Colouring Matters of Australian Plants. II— Naphthaquinones from *Diospyros hebecarpa* R. G. Cooke and H. Dowd. *Australian J. Sci. Research*, **5A**, 760–767 (1952); **III— Synthesis of 7-Methyljuglone and Related Compounds.** *Ibid.*, **6**, 53–57 (1953): *Chem. Abs.*, **48**, 2022 (25 Feb. 1954).

The colouring matters of *Diospyros hebecarpa* are 5-hydroxy-7-methyl-1:4-naphthaquinone and 1:4-dioxo-5-hydroxy-7-methyl-1:2:3:4-tetrahydronaphthalene. Their synthesis is described. C. O. C.

Colouring Matter in Turmeric. K. R. Srinivasan. *Current Sci. (India)*, **21**, 311–312 (1952): *Chem. Abs.*, **48**, 1099 (25 Feb. 1954).

Diferuloylmethane and two other pigments, probably (*p* - hydroxycinnamoyl) - feruloylmethane and bis(*p*-hydroxycinnamoyl)-methane, were separated from a benzene extract of turmeric by chromatography on silica gel. C. O. C.

Colouring Matters of the Aphididæ. IX— Some Reactions of Extended Quinones. B. R. Brown and A. R. Todd. *J.C.S.*, 1280–1285 (April 1954).

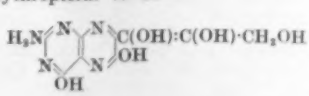
Perylene-3:10-quinone reacts with *cyclo*hexylamine, piperidine, and aq. NaOH in presence of air to give respectively 1-*cyclo*hexylamino-, 1-piperidino-, and 1-hydroxy-perylene-3:10-quinone. 2:11-Dihydroxyperylene-3:10-quinone is also shown to yield a monopiperidino deriv. The data support the current view that erythroaphins are deriv. of perylene-3:10-quinone. H. H. H.

Colouring Matters of the Aphididæ. X— Preparation and Properties of 4:9-Dihydroxyperylene-3:10-quinone. A. Calderbank, A. W. Johnson, and A. R. Todd. *J.C.S.*, 1285–1289 (April 1954).

The compound described in the literature as perylene-3:4:9:10-diquinone is now shown to be 4:9-dihydroxyperylene-3:10-quinone. The similarity of its ultraviolet-absorption spectrum to that of erythroaphin-*fb* confirms that this ring system is the chromophore of the aphin. H. H. H.

Pigment of the Wings of Butterflies. XVI—Erythropterin. R. Purrmann and F. Eulitz. *Annalen*, **559**, 169–174 (1948): *Chem. Abs.*, **48**, 3985 (10 April 1954).

Evidence is given for the structure of the orange-red pigment, erythropterin to be—



C. O. C.

Precursors of the Red Colour in Leather. W. E. Hillis. *J. Soc. Leather Trades Chem.*, **38**, 91–102 (March 1954).

Tannins from the bark and the wood of certain *Eucalyptus* species are heated with propanol and HCl to give anthocyanidins, and it is suggested that the leucoanthocyanins from which the anthocyanidins originated are the precursors of the red colour in leather tanned wth these materials. J. W. B.

Cyanopsin, A New Pigment of Cone Vision. G. Wald, P. K. Brown, and P. H. Smith. *Science*, **118**, 505–508 (1953): *Chem. Abs.*, **48**, 3412 (25 March 1954).

A new visual pigment, cyanopsin, has been synthesised by combining chicken cone opsin and retinene$_2$.

C. O. C.

Ceramic Pigments and Opacity Agents. I—The Pinks of Chromium and Manganese. A. B. Monzó and F. C. Landete. *Ion*, **13**, 383–391 (1953): *Chem. Abs.*, **48**, 3653 (25 March 1954).

Results of many experiments show that: kieselguhr is a better source of SiO_2 than clay; CaF_2 brings out a superior pink even under unfavourable oxidising conditions; Cr_2O_3 should be introduced as a soluble salt; coprecipitation of the components of the mix is, when possible, superior to mechanical mixing; the Cr_2O_3–SnO_2 series gives the best colours; the Cr_2O_3–Al_2O_3 and the Cr_2O_3–MnO pigments are inferior to the others but have considerable economic importance. C. O. C.

250 Years of Prussian Blue. F. Fritz. *Deut. Farben-Z.*, **8**, 7–8 (1954): *Chem. Abs.*, **48**, 4855 (25 April 1954). Historical. C. O. C.

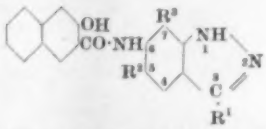
Iron Oxide Pigments from Ferrous Chloride. J. Kunzweiler. *Deut. Farben-Z.*, **7**, 442–443 (1953): *Chem. Abs.*, **48**, 2387 (25 Feb. 1954).

Light ochre to reddish brown pigments are produced by varying the conditions under which aqueous ferrous chloride is treated with calcium chloride. C. O. C.

PATENTS

Azoic Coupling Components Derived from 6-Aminoindazoles. ICI. *BP* 707,897

Arylamides



(R^1 R^2 R^3 = H or Alk of <4C), made by condensing 3-hydroxy-2-naphthoic acid with 6-aminoindazole or its appropriate alkyl derivatives, have good substantivity for cotton, and couple with suitable diazotised amines to give a range of reddish-oranges, scarlets, reds, bordeaux and garnets. They dissolve readily in dil. aq. alkalis to give solutions of good stability. Thus 6-aminoindazole is condensed with 3-hydroxy-2-naphthoic acid by refluxing in toluene and adding PCl_3. The product is dissolved in dil. aq. NaOH, salt is added, and cotton yarn in package form is impregnated with the solution. Development in diazotised [2:5-dichloroaniline gives a bright scarlet. E. S.

Azoic Dyeings on Leather. FH. *BP* 707,884

Soln. of mixtures of coupling components and antidiazotates in caustic alkalis and containing also water-soluble solvents, wetting agents, protective colloids, etc., may be applied to leather by immersion methods or by brushing or spraying; treatment with a weakly acid solution then develops a well-penetrated azoic colour. If the coupling component contains a carboxy or sulpho group, dyeings of good rubbing fastness are obtained. Thus leather which has been first tanned with a vegetable extract and then chrome-tanned is immersed for 1 min. in a soln. containing 3-hydroxy-2-naphtho-*m*-carboxyanilide, the antidiazotate from 2-amino-5-chlorotoluene, NaOH, ethyl alcohol, sodium sulphoricinoleate, and sodium chromate. After squeezing, the well-penetrated red colour is developed by treatment in dil. aq. acetic acid containing salt at 40°C. for 30 sec. followed by standing for 30 min., rinsing, and drying. E. S.

Metal(Chromium)-complex Monoazo Wool Dyes. Ciba. *BP* 707,016

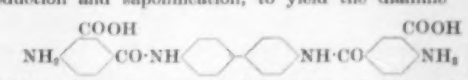
The Cr complexes of monoazo dyes *o*-aminophenol (or preferably *o*-alkoxyaniline) → 8-chloronaphtholsulphonic acid containing 1 atom Cr to 1 mol. azo compound are red, violet, or blue wool dyes. Thus the monoazo compound *o*-anisidine → 8-chloronaphthol-3:6-disulphonic acid is heated at 130–135°C. in a lead-lined autoclave with $Cr(OH)SO_4$. The product dyes wool blue-violet from a bath containing H_2SO_4. E. S.

Metal-complex Monoazo Dyes for Anodised Aluminium, etc. Ciba. *BP* 703,949

The copper and cobalt complexes of the monoazo dyes 2-aminophenol-4:6-disulphonic acid → a 3-methyl-1-phenyl-5-pyrazolone give yellows of good light fastness on anodised aluminium, and also dye animal fibres. Thus the monoazo compound 2-aminophenol-4:6-disulphonic acid → 3-methyl-1-*o*-chlorophenyl-5-pyrazolone is heated at 70–75°C. with aq. cobalt sulphate in presence of sodium acetate. The product dyes anodised aluminium golden yellow. E. S.

Metallisable Direct Cotton Azo Dyes. S. *BP* 708,681

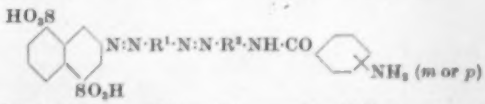
Aromatic primary mono- and di-amines are acylated with 4(or 3)-carboxyalkyl-3(or 4)- nitrobenzoyl chloride, followed by reduction of nitro to amino groups, and saponification of the ester group to COOH. The amino compounds so produced are di- or tetr-azotised and coupled with suitable *o*-coupling phenols, naphthols, pyrazolones, etc., to give direct cotton dyes, which may be metallised in substance or on the fibre. Thus benzidine is treated in an organic solvent with 3-carboxymethyl-4-nitrobenzoyl chloride in presence of an acid-binding agent, followed by reduction and saponification, to yield the diamine



which is tetrazotised and coupled with 2 mol. of 3-methyl-1-*p*-sulphophenyl-5-pyrazolone. The disazo compound so formed dyes cotton yellow by the after-coppering process. E. S.

Metallisable Direct Cotton Trisazo Dyes. Ciba. *BP* 708,663

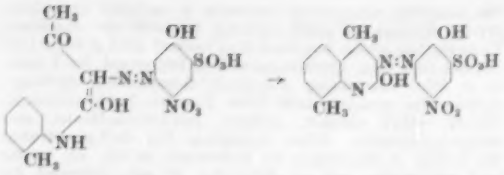
Direct cotton dyes which may be metallised (particularly with Cu salts) in the dyebath and/or on the fibre are made by diazotising an amino-disazo compound—



(R^1 and R^2 = residues of benzenoid middle components) and coupling with an *o*-hydroxybenzoic acid. Thus the disazo compound 2-naphthylamine-4:8-disulphonic acid → *m*-toluidine → *m*-toluidine is *N*-acylated with *p*-nitrobenzoyl chloride, and the NO_2 group reduced to NH_2. The product is then diazotised and coupled with salicylic acid. The resulting dye gives dischargeable oranges on cotton by an after-coppering process. E. S.

Metallisable Azo Carbostyril Dyes. American Cyanamid Co. *USP* 2,640,825

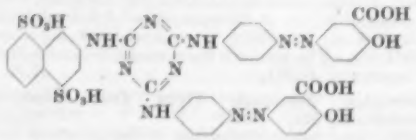
Metallisable *oo'*-dihydroxyazo dyes containing a carbostyril residue are made by cyclisation of azo dyes of the type *o*-aminophenol → acetoacetarylide. Thus the yellow monoazo compound 2-amino-4-nitrophenol-6-sulphonic acid → acetoacet-*o*-toluidide is heated at 60°C. for 1 hr. in conc. H_2SO_4, when the carbostyril compound is formed as follows—



It dyes wool red-orange, and may be converted into a metal complex by refluxing in aq. chromium acetate.
E. S.

Dischargeable Direct Cotton Disazo Dyes. Ciba. *BP* 707,488

Disazo dyes of the type—



dye cotton yellow and are dischargeable to a good white with sodium formaldehyde-sulphoxylate. The dye of the above formula for example is made by condensing cyanuric chloride, first with 2-naphthylamine-4:8-disulphonic acid in presence of aq. sodium acetate at 0°c., then with 1 mol. of 4-amino-4′-hydroxyazobenzene-3′-carboxylic acid (I) at 30–35°c., and finally with a second mol. of I at 90–95°c.
E. S.

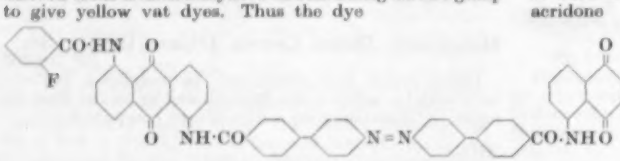
Non-diffusible Azo, Anthraquinonoid, Triphenylmethane, etc. Dyes for Photographic Emulsion Layers. Eastman Kodak Co. *USP* 2,632,004

Dyes which do not tend to migrate from a photographic emulsion layer are made by *O*-acylation of azo, anthraquinonoid, triphenylmethane, etc. dyes containing an hydroxyalkyl group by means of polymers containing carboxylic halide or anhydride groups.

Thus the monoazo dye sulphanilic acid → *N*-ethyl-*N*-hydroxyethylaniline is heated in pyridine with a soln. of polyacrylyl chloride in acetone. The Na salt of the product is dispersible in water and compatible with gelatine, from which it does not diffuse into an adjacent orthochromatic emulsion coating.
E. S.

Fluorobenzoylaminoanthraquinone Vat Dyes containing an Azo Group. Ciba. *BP* 706,737

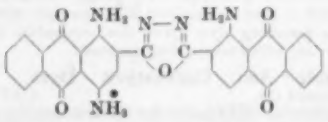
Two mol. of 1-amino-5-(*o*-fluorobenzoylamino)-anthraquinone are reacted with one mol. of an acylating agent derived from a dicarboxylic acid containing an azo group to give yellow vat dyes. Thus the dye



is prepared by heating azodiphenyl dicarboxylic acid with 1-amino-5-(*o*-fluorobenzoylamino)-anthraquinone at 100°c. to boiling for 2 hr. in trichlorobenzene, in presence of thionyl chloride and pyridine.
R. K. F.

Dianthraquinonyl-1:3:4-oxadiazoles—Vat Dyes. ICI. *BP* 705,920

The compound—



is acylated at the N marked * to produce violet dyes which can be applied at high temperatures. Thus 2-(1-amino-2-anthraquinonyl)-5-(1:4-diamino-2-anthraquinonyl)-1:3:4-oxadiazole is heated at 130°c. for 12 hr. with benzoyl chloride in nitrobenzene containing pyridine.
R. K. F.

Dianthraquinonyl-1:3:4-oxadiazole—Vat Dyes. ICI. *BP* 705,928

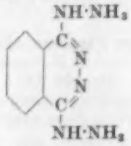
N:*N*′-Bis-(1-aminoanthraquinonyl-2-carbonyl)-hydrazine (from 1-aminoanthraquinone-2-carbonyl chloride and hydrazine) is heated at 95–100°c. for 30 min. in 0·5%–5% oleum to give the corresponding 1:3:4-oxadiazole—a red vat dye.

BP 705,929

The process is modified to obtain tri- and tetra-amino dianthraquinonyloxadiazoles. Thus *N*:*N*′-bis-(1-amino-4-*p*-toluene sulphonamidoanthraquinone-2-carboxyl)-hydrazine is heated for 4 hr. at 95–100°c. with 2% oleum. The product, in which the *p*-toluene sulphonyl groups have been hydrolysed off to give 2:5-di(1′:4′-diamino-2′-anthraquinonyl)-1:3:4-oxadiazole, is separated by diluting with water to 80% H_2SO_4.
R. K. F.

1:4-Dihydrazinophthalazines. CFM. *BP* 707,337

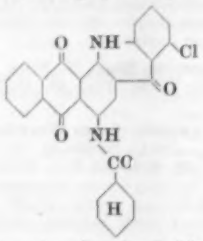
1:4-dihydrazinophthalazines are prepared by treating phthalodinitriles with hydrazine. Thus 1:4-dihydrazinophthalazine—



is obtained by heating phthalodinitrile in dioxan with a solution of hydrazine hydrate in 50% aq. acetic acid for 3 hr. at 95°c. The products are intermediates for dyes.
R. K. F.

8-Chloro-2-hydroaroylamino-3:4-phthaloylacridones—Vat Dyes. CFM. *BP* 707,576

A 1-amino-4(2′-carboxy-3′-chlorophenyl)-aminoanthraquinone is cyclised to the corresponding chloroacridone and the amino group acylated to introduce a hydroaroyl group. The products are grey vat dyes. Thus the dye—



is obtained by treating 2-amino-8-chloro-3:4-phthaloylacridone (from 1-amino-4-bromoanthraquinone-2-sulphonic acid and 6-chloro-2-aminobenzoic acid) with hexahydrobenzoyl chloride in monochlorobenzene for 12 hr. at 120°c.
R. K. F.

Sulphur-containing Vattable Phthalocyanines. Basf. *BP* 707,710

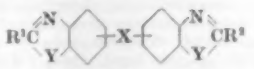
Sulphur is introduced during the formation of a phthalocyanine from aromatic *o*-dinitriles or similar derivatives and a finely divided metal or metallic compound, using ≯ 24 at. of metal per 4 mol. of phthalocyanine-forming compound. The products are blue dyes capable of being dyed from a hydrosulphite or sulphide vat. Thus, all parts being by weight, a mixture of phthalodinitrile (51), anhyd. cobalt chloride (15) and sulphur (9·6) is heated at 180–200°c. The cooled melt is powdered and extracted first with aq. NaOH and then with dil. HCl.
R. K. F.

Isothiouronium Dye Compositions. ICI. *BP* 707,557

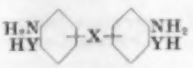
The stability of powders containing isothiouronium dyes, e.g. the product obtained by condensing copper tris-chloromethyl-phthalocyanine with tetramethyl-thiourea, is improved by admixing 0·1 to 5 parts per 1 part dye of a solid water soluble acidic substance, e.g. sodium bisulphate or tartaric acid.
R. K. F.

Fluorescent Brightening Agents. CFM. *BP* 708,651

Compounds of formula —



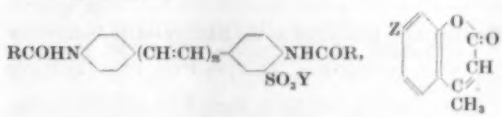
(R^1 and R^2 = same or different, H, Ar or aralkyl; Y = O, S or NH; X = a direct linkage or –CH:CH–; the aromatic nuclei may contain SO_3H groups) having good affinity for cellulose and forceful fluorescence are obtained by condensing compounds of formula —

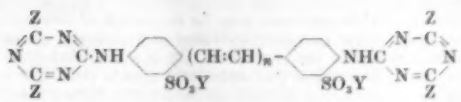


with aldehydes or acids or their functional derivatives. Thus *oo'*-dihydroxybenzidene (216 parts by wt.) is heated with benzoylchloride (1000) at 250°c. for 5 hr. with continuous stirring. Excess benzoylchloride is then distilled off and the residue boiled with methanol (1000). The mass is filtered hot and the residue boiled with benzene (2000), then cooled, filtered and washed with benzene. On recrystallising from chlorobenzene colourless laminæ are obtained. Cotton treated in a 1:40 liquor at 30°c. for 30 min. with a solution containing 0·025 g. per litre of the above product and 0·597 g. of formic acid becomes a beautiful white. C. O. C.

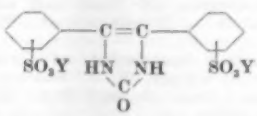
Fluorescent Brightening Agents for improving Whites of Photographic Prints. General Aniline. *BP* 709,111

Fluorescent brightening agents having one of the following formulæ —





or



(R = phenyl; X = Alk or phenyl; Z = OH, alkylamino, hydroxyalkylamino, arylamino, *cyclo*alkylamino or *N*-morpholinyl; Y = H or a salt forming group; n = 0 or 1), e.g. 4:5-diphenylimidazolidone— (2)-*pp'*-disulphonic acid, have high affinity for cellulose, gelatin or other colloids, e.g. gelled polyvinyl alcohol, on which they are fast to washing. C. O. C.

Cadmium Red. Glidden Co. *USP* 2,643,196

Loss of selenium during calcination, use of low calcinary temperatures, and greater uniformity and control of hue from batch to batch are obtained if the crude cadmium red has had an oxidic cadmium compound coprecipitated with it, e.g. by striking a solution of a cadmium salt with an aqueous alkaline sulphide solution containing selenium and an oxidic compound of an alkali or alkaline earth metal. C. O. C.

Carbon Black. Phillips Petroleum Co. *USP* 2,641,533

Carbon black has its pH valve reduced by treating it with an oxyhalide in presence of a diluent gas.

USP 2,641,534

An improved burner for making furnace black.

USP 2,641,535

Furnace black has its pH lowered and is made more hydrophilic by treating it with hot air containing a little SO_3. C. O. C.

Calcined Channel Black. J. M. Huber Corpn. *USP* 2,643,182

Pelleting of Carbon Black. Phillips Petroleum Co. *USP* 2,642,343

Clays, Precipitated Chalk, etc. English Clays Lovering Pochin & Co. *BP* 706,429

Pigments such as clays, precipitated chalk, etc. of particle size 10μ. or less have the proportion of particles $< 1\mu$. increased by passing a filter cake of the pigment containing < 4°c. moisture through a pugmill so made that it absorbs energy of > 8 h.p. hours per ton of output. C. O. C.

Cuprous Sulphide as Black Pigment for Anti-fouling Paints. Robertson Tham. *BP* 708,147

A black pigment particularly suitable for use in anti-fouling paints is obtained by reacting finely divided sulphur in less than stoichiometric amount with finely divided metallic copper suspended in slightly acid aqueous medium, separating the cuprous sulphide from the copper, drying and grinding. C. O. C.

Kaolin of Fine Particle Size. J. M. Huber Corpn. *BP* 706,537

Kaolin or kaolinitic clay containing less than 40% Al_2O_3 is treated at $\leqslant 120$°c. with $\leqslant 25\%$ of its wt. of caustic alkali dissolved in water, to disintegrate it so that no particle is $> 0{\cdot}5\mu$. C. O. C.

Finely Divided Metal Oxides and Silica. Säurefabrik Schweizerhall. *BP* 708,970

An improved method of the high temperature decomposition of metal chloride vapours with oxygen containing gases. C. O. C.

Titanium Dioxide. DuP. *BP* 707,560

$TiCl_4$ and a little $AlCl_3$ are treated at $\leqslant 800$°c. with an oxygen-containing gas in presence of water (0·1–5·0% by volume of the gas) for 0·01–1·0 sec. and immediately cooling to < 600°c. The product has pH 6·5–7·5, and is readily ground. Compared with the product obtained by *USP* 2,488,439 it is softer and has better covering power, tinting strength, colour and fastness to light, baking and pigment chalking. C. O. C.

"Naphthanil" Coupling Components— Relationship between Chemical Structure and Substantivity for Cellulose (VIII p. 318).

Neutral Premetallised Dyes (VIII p. 319).

Polarographic Method of Analysis in Control of Production in the Aniline Dye Industry (XIV p. 328).

V— PAINTS; ENAMELS; INKS

Historical Outline of the Development of Paints. J. H. de Vlieger. *Verfkroniek*, **26**, 202–205, 210, 229–233, 255–257, 287–290, 316–318, 340–341 (1953): *Chem. Abs.*, **48**, 3703 (25 March 1954).

An account of chemical and industrial developments in paint during the 19th century; 118 references. C. O. C.

Effect of Surface-active Agents on Pigment-Vehicle Systems. R. Denning and F. K. Daniel. *Offic. Dig. Federation Paint & Varnish Production Clubs*, **26**, 37–44 (1954): *Chem. Abs.*, **48**, 4855 (25 April 1954).

Flow-point determinations with three vehicles, six pigments and five surface-active agents show that dispersibility depends neither on the pigment alone, nor on the vehicle alone nor on the surface-active agent alone, but on the combined effect of all three. C. O. C.

Microstructure of Paint Films. E. G. Bobalek, L. R. Lebras, A. S. Powell, and W. von Fischer. *Ind. Eng. Chem.*, **46**, 572–577 (March 1954).

The silver-silica and polyvinyl alcohol-silica techniques for the preparation of specimens for electron microscopy are described, and 25 electron micrographs are reproduced in illustration of the discussion. Gloss paints show smooth surfaces overlaid with a film of clear binder, and occasional pigment particles do not interrupt the film surface. As the pigment : binder ratio is increased to give semi-gloss and, finally, matt finishes, the surface becomes rougher, and more broken by pigment particles. Differences in gloss amongst paints of apparently identical formulation cannot wholly be explained in terms of degree of dispersion of the pigment. Evidence is discussed which

suggests that in the paints of lower gloss there are pigment particles which are not effectively wetted out by the binder medium, and these tend to float to the surface of the film; such particles are absent from or at a min. in paints of good gloss. Aggregation does play a part, and primary pigment particles and very small flocculates tend to settle to the surface of adhesion, whilst larger aggregates float to the paint–air interface, where they cause roughness; there is less roughness due to pigment at the adhesion interface than at the upper surface. This effect is accentuated in thick films, which provide longer times for fractionation of the pigment. The effect of "blushing" phenomena on surface appearance has not yet been adequately investigated. The application of electron microscopy to mixed pigment systems is briefly discussed by reference to a series of tinted enamels. Examination of stripped films suggests that only a small fraction of a substrate interface is bound strongly to an organic coating film. J. W. D.

PATENTS

Printing Ink of the Oxidising Type containing a Polymerised Olefin. Universal Oil Products Co. *USP* 2,640,782

An ink having greater flow, greater rub and scratch resistance, and penetration and which is quicker drying contains a hydrocarbon drying oil e.g. a high mol. wt. polyolefine, as the principal component of the vehicle. C. O. C.

Printing Ink of Improved Drying Properties. National Printing Ink Research Institute. *USP* 2,640,783

Cobalt borate, precipitated in presence of a wetting agent, used as a drier greatly reduces the severe loss of drier effectiveness shown by many printing inks. The effect is even more pronounced if it is used in conjunction with a small amount of cobalt linoleate. C. O. C.

Printing Ink for Asbestos-Cement Bases. Ruberoid Co. *USP* 2,643,191

An ink which when applied to a freshly formed sheet of asbestos-cement does not smear, blend, drift or bleed and which does not inhibit drying of the cement, consists of pigment and hydraulic or Portland cement dispersed in a vehicle made up mostly by an oil or liquid hydrocarbon which does not inhibit hydration of the cement. An aluminium soap may also be present as a bodying agent. C. O. C.

Film-forming and Drying Composition. Titan Co. *BP* 707,916

A composition which quickly dries to yield a film resistant to organic solvents, alkali and water consists of a cellulosic derivative containing 0·2–0·8 free OH groups per glucose unit and an alkyl titanate dissolved in a volatile solvent containing a monohydric alcohol. C. O. C.

Paints. Baker Perkins. *BP* 709,020

The pigment is precipitated as usual, formed into a slurry, heated to about 50°c. and then mixed with a vehicle at the same temperature in a colour flushing vessel. Mixing is done under atmospheric pressure until a homogeneous mix is obtained and the precipitation water has been removed after which mixing is continued under vacuum at a temperature which ensures evaporation of the remaining water. The necessary thinners are then added from a supply at atmospheric temperature, preferably the flushing vessel, to produce the finished paint ready for tapping off. C. O. C.

Lacquers on Enamels yielding Translucent Coatings of Metallic Appearance. DuP. *USP* 2,642,404

A coating composition yielding tough and durable films having a metallic appearance of great depth and brilliance comprises a vehicle and as pigment a mixture of (1) the Ni complex of the dye *p*-chloroaniline→ 2:4-dihydroxyquinoline, (2) sufficient of a transparent pigment substantially complementary in colour to the foregoing Ni complex to form a grey when they are mixed together, (3) a coloured tinting pigment, and (4) finely-divided aluminium flake. Suitable transparent pigments are the lake of dichloroisodibenzanthrone on Ba rosinate, copper phthalocyanine, phthalocyanine green, or the thio-indigo vat dye Indo Maroon. C. O. C.

Protective and Decorative Coatings on Aluminium or Aluminium-base Alloys. American Chemical Paint Co. *BP* 708,937

Aqueous solutions containing phosphate and/or arsenate ions, fluoride ions, chromate ions and/or sulphate ions present in amounts stoichiometrically equivalent to fluoride 0·5–8·99 (g. per litre), CrO_3 7·4–66·6, PO_4 (including arsenate calculated as PO_4) 2·0–285·0, chloride and/or sulphate (both calculated as NaCl) 1·0–5·0. The ratio of $F:CrO_3$ is 0·015–0·135 and the pH of the solution is 1·0–3·2. C. O. C.

Mat Finish Enamels for Aluminium. DuP. *USP* 2,642,364

Azine Dyes as Temperature Indicators in Cellulose Ester-Furfural Coating Compositions. J. de la Valdene. *BP* 709,304

When applying coatings based on cellulose esters and furfural, azine dyes, e.g. Malachite Green, are added to the composition to act as indicators, the dyes being decolourised when the correct polymerisation temperature has been reached during baking. Preferably they are used in conjunction with another dye which is unaffected by the temperature reached. C. O. C.

VI— FIBRES; YARNS; FABRICS

Determination of the Specific Electrical Resistance of Fibres. K. Götze, W. Brasseler, and F. Hilger. *Melliand Textilber.*, **34**, 1142–1143 (Dec. 1953).

If a charged electrometer (with or without a condenser in parallel) is discharged via a textile material, the time taken depends on the resistance of the latter. The theory involved is given, and, as examples, the specific resistances of an acetate and a viscose rayon are determined to be $2{\cdot}66 \times 10^{11}$ and $4{\cdot}1 \times 10^{6}$ ohm-cm. respectively. H. E. N.

Resistivity— One Clue to the Electrostatic Behaviour of Fabric. N. A. Teixeira and S. M. Edelstein. *Amer. Dyestuff Rep.*, **43**, P195–P199, P208 (29 March 1954).

Fundamental principles of static formation on textiles, mechanisms whereby static can be dissipated, and measurements to which materials may be subjected to assess both the tendency to become electrified and also the effect of antistatic agents are discussed. It is concluded that in evaluating the efficacy of antistatic agents practical mill exposure is more important than laboratory measurements. J. W. B.

Electrical Resistance of Wool and other Textile Materials. M. L. Wright. *J.S.D.C.*, **70**, 120 (March 1954).

Practical Partial Acetylation of Cotton. E. M. Buras, A. S. Cooper, E. J. Keating, and C. F. Goldthwait. *Amer. Dyestuff Rep.*, **43**, P203–P208 (29 March 1954).

Batch and continuous processes for acetylating cotton are described. Fabric is handled on stainless-steel dye jigs or on a newly developed continuous range, yarns are treated in package form or as continuous warp, and raw stock is handled in a raw-stock dyeing machine. Except when treating raw stock, a preliminary desize and an alkali boil are necessary, after which, for batch processing, there are three stages— activation, esterification, and washing. In the first, the cotton is soaked for from 1 hr. to overnight in glacial acetic acid, and squeezed. Esterification is effected by treating with a mixture of 85% acetic acid, 15% acetic anhydride, and 0·1–0·2% perchloric acid catalyst, the total being about 8 × wt. of cotton. The working temp. is about 68°F., and cooling is necessary to dissipate heat generated. Control tests are necessary to check the progress of acetylation, as, for apparently trivial reasons, differences arise from batch to batch. Speed of reaction can be controlled by piecemeal addition of acetic anhydride. Finally the fabric is washed and dried in the conventional manner. In continuous processing the activation is for 2 min. only, at 180°F.; the material is cooled and passed through a bath of 1–2% of perchloric acid in acetic acid, which serves to apply a suitable amount of catalyst. This occupies 1 min., and is followed by passage through 40% acetic anhydride in acetic acid, at 70°F. for 1–3 min., after which the fabric is washed and dried. J. W. B.

New Fibres with the Structural Elements of Natural Cellulosic Fibres. J. Compton. *Amer. Dyestuff Rep.*, **43**, P103–P111 (15 Feb. 1954).

The physical and chemical structures of cotton are discussed, and the advantageous properties of cotton tabulated. It is claimed that partly cyanoethylated cotton, produced by reaction with acrylonitrile, enhances these properties by increasing resistance to heat, abrasion, and microorganisms, and by increasing dye affinity.

J. W. B.

Amount of Non-Freezing Water in Swollen Cellulosic Fibres. N. I. Klenkova and N. I. Nikitin. *J. Appl. Chem. U.S.S.R.*, **27**, 171–181 (Feb. 1954).

The amount of non-freezing (i.e. strongly attached) water present in various swollen cellulosic fibres is determined by a calorimetric method based, in principle, on that of Magne *et al.* (see J.S.D.C., **64**, 120 (March 1948)), freezing temperatures varying from $-5°$ to $-21°$C. being used (duration of freezing treatment 3·5 hr.). The results are closely parallel to those derived from sorption isotherms and from rates of hydrolysis, and they may be related in a similar way to the degree of accessibility of fibre molecules to reagents. Some typical contents of non-freezing water (% on wt. of dry fibre for a freezing temp. of $\sim -5·5°$C.; water absorbed from vapour) are — ramie, 12·5; untreated linters, 11·9; mercerised linters, 26·4; viscose rayon, 35·9; hydroxyethylated linters, 46·6–56·7, depending on the degree of substitution (3·8–14·6% of $O{\cdot}C_2H_4{\cdot}OH$). Further experiments show the great effect of drying on cellulosic materials that have been swollen by a chemical treatment and then washed (e.g. a sulphate-cellulose pulp taken after chlorination without being dried contains 53·6% of non-freezing water ($-5·5°$C.) whereas after being dried and then re-swollen in water it contains 16·5% of non-freezing water). In all cases the content of non-freezing water is greater when swelling occurs in liquid water than when it results from the absorption of vapour (exposure to vapour from 5% H_2SO_4 for 30 days). It is considered that in the former case additional opening of the fibre structure occurs, rendering further parts of the chain molecules accessible to the water.

A. E. S.

Viscose Staple Research. H. Brandenburger. *Melliand Textilber.*, **34**, 264–266 (April), 405–406 (May), 499–500 (June), 617–618 (July), 823–824 (Sept.), 1027–1028 (Nov.), 1118–1120 (Dec. 1953).

An account, with 93 references, of research completed in 1943–1945. The effects of the degree of polymerisation alone and in combination with the other variables of the manufacturing process on the properties of the viscose and the rayon prepared from it were studied. The results are discussed in detail under the headings— preparation of the starting material, properties of the alkali cellulose and of the viscose, and the spinning process.

H. E. N.

Electron-microscopic Study of the Fine Structure of Viscose Rayon. P. Kassenbeck. *Bull. Inst. Textile France*, (43), 43–52 (Dec. 1953).

New methods for the preparation of fibre sections are described. The technique makes it possible to study the "skin" and "core" layers of viscose rayon fibres and to follow the effects of solvent on the fibre surface. It is claimed that the fibres have a cuticle differing in structure from the "skin" which can be observed under the optical microscope.

G. L.

Particle Properties of the Crystalline Regions of the Cellulose Fibre. W. Kast. *Z. Elektrochem.*, **57**, 525–530 (1953): *Chem. Abs.*, **48**, 4217 (10 April 1954).

Measurements of the crystalline region of cellulose are in good agreement by both the X-ray and the volumetric methods. Therefore it seems that transition from the crystalline to the non-crystalline regions is only slightly blurred. Comparison of the crystallinities with the accessibilities (measured by water absorption) shows that the regions deemed to be crystalline on the basis of X-ray interferences or their increased density are identical with those which are only moderately reactive as far as water absorption and $H \rightarrow D$ exchange are concerned. Reasonable values for the diameters of these regions are obtained when their capability of being attacked is explained as resulting from a surface reaction (with cellulose II hydration of the lattice must be reckoned with). The crystalline regions of cellulose I and II behave, therefore, to a great extent like particles which are separated fairly sharply from the non-crystalline material. This may result from the process of growth or may be caused by the cellulose not being dissolved to molecular dispersity in the spinning solutions. Moreover, the far-reaching agreement of crystallinities of the regenerated fibres indicates that a certain part of the molecular chains from the crystalline regions of the native fibres remains in the solution in a position ready to crystallise. In the synthetic fibres, prepared from high polymer melts, a running transition of crystalline or non-crystalline regions seems to occur.

C. O. C.

Membrane Potentials for Keratin and Cellophane and the Meyer–Teorell Theory. M. L. Wright. *J. Physical Chem.*, **58**, 50–53 (1954): *Chem. Abs.*, **48**, 3758 (10 April 1954).

Membrane-potential measurements are merely ionic-transport number determinations for diffusion in the polymer and no further quantative information concerning the system can be deduced from the membrane-potential data alone. If the Meyer-Teorell theory is applied to analysing membrane-potential measurements to obtain the ionic-mobility ratio the result will be defective as the internal diffusion potential which can arise from difference in ionic concentration gradients inside the membrane and which may exist even when the mobilities are equal, is ignored. The boundary and internal diffusion potential components, calculated from sorption data, agree with experiments and show that the second potential often forms a large part of the total potential. Thus any data deduced by use of the original Meyer–Teorell analysis are of doubtful value. To characterise a membrane sorption data are also required and then the ratio of the ionic mobilities can be calculated, and from this ratio, the individual mobilities can be obtained by use of membrane conductivity data.

C. O. C.

Disulphide Bond Reactivity in Wool— Formation of Thiol Radicals on Exposure to Light. G. H. Crawshaw and J. B. Speakman. J.S.D.C., **70**, 81–82 (Feb. 1954).

On exposure to diffuse light, stringently de-aerated solutions containing methacrylic acid and sulphuric acid undergo polymerisation in a few hours at 25°c. in presence of ethyl disulphide, cystine, or intact wool, but not in their absence. The disulphides (including wool) are ineffective in the dark, and it is concluded that polymerisation is initiated by the thiol radicals which are produced by homolytic fission of the disulphide bonds under the influence of light—

$$R{\cdot}S{\cdot}S{\cdot}R \underset{\text{dark}}{\overset{h\nu}{\rightleftharpoons}} 2\,R{\cdot}S^{\bullet}$$

Adsorption of Water by Wool. IV— Influence of Combined Acid on the Affinity of Wool for Water. C. H. Nicholls and J. B. Speakman. *J. Textile Inst.*, **45**, T267–T271 (March 1954).

Purified wool is brought to equilibrium in solutions of free Orange II and of sulphuric, oxalic, phosphoric, mono- and di-chloroacetic, naphthalene-β-sulphonic, and picric acids. The water adsorption isotherms of the various samples of acid-containing material are determined by exposure to the vapour from H_2SO_4 solutions of varying densities. It is found that the water adsorption capacities of the acid-containing wools are less than that of untreated wool, except in certain cases where the humidity approaches saturation. The theoretical view that anions with a high affinity for water-adsorbing sites in the wool will have the greatest effect on subsequent water adsorption is supported by the fact that picric acid, with affinity for –OH and =NH groups, has the greatest effect in reducing regain. Sulphuric acid, which has heavily hydrated anions with a low affinity for wool, reduces the regain only slightly. Only a small part of the fall in regain is due to back-titration of the $-COO^-$ ions of salt linkages.

J. W. B.

Action of Bromine Water on Wool. M. Leveau, A. Paulsot, and N. Cebe. *Bull. Inst. Textile France* (42), 7–9 (Oct. 1953).

Bromine water reacts with wool in an asymmetrical manner. When fibres stained with Methylene Blue were microscopically examined, it was found that part of the

cortex was considerably swollen and that the Allwörden effect occurred preferentially where swelling was small. It is claimed that the results are in agreement with previous experimental evidence on the subdivision of the cortex of wool. G. L.

Reaction Rate of Bromine Water on Wool Fibres. M. Leveau and N. Cebe. *Bull. Inst. Textile France*, (43), 33–41 (Dec. 1953).

A study of some variables affecting the time necessary for Allwörden's bubbles to appear on wool fibres immersed in bromine water. Acidic pretreatment of the wool or more concentrated bromine water increases the reaction rate, but alkaline wool requires more time. The pH of the bromine water is not important as long as it remains acidic. The effect of metallic ions added to the bromine water on the speed of reaction is specific: thus mercuric ions accelerate, Cu ions retard, whilst Mg ions have no influence. G. L.

Effect of Thioglycollic Acid on the Mechanical Properties of Hair. W. Fuhrmann. *Melliand Textilber.*, **34**, 1022–1025 (Nov. 1953).

From load–elongation diagrams it is shown that treatment of hair with a soln. of thioglycollic acid (I) first slightly weakens the elastic cell-substance, but then suddenly the plastic material between the cells is attacked and rapid weakening sets in. Hairs only slightly weakened with I are further weakened when treated with a soln. of H_2O_2 or when immersed in oxygen-free water. Hair more greatly softened with I is hardened on replacement of I by water, and further hardened on the substitution of a soln. of H_2O_2 for the water. Graphs also illustrate the superiority of mono- over di- and tri-ethanolamine and over NaOH as the alkali used in conjunction with I. H. E. N.

Recoverability of Hairs. W. Fuhrmann. *Faserforsch. und Textiltech.*, **5**, 22–27 (Jan. 1954).

Extension–relaxation cycles on hairs are analysed with the aid of the author's fibre model (see J.S.D.C., **69**, 98 (March 1953)) in order to provide a measure of the permanent effect of various processes (drying under tension, and treatment with acids, enzymes, oxidising agents, reducing agents, and sulphite–pyridine solutions) on the mechanical properties of the fibre. A. E. S.

Adsorption Birefringence in Cellulose Acetates. A. Möhring. *Faserforsch. und Textiltech.*, **5**, 105–111 (March 1954).

The birefringence of wet-spun cellulose triacetate fibres, which, like that of all cellulose triester fibres hitherto investigated, is negative, becomes positive when the fibres are swollen in toluene or xylene, but not when they are swollen in benzene, chlorobenzene, methanol, or ethanol. The reversal of sign does not occur in cellulose triacetate obtained by acetylation, with retention of fibre form, of cotton or viscose (ordinary or Lilienfeld) fibres, but approximately quantitative measurements (of retardation with the aid of a Berek compensator) show that the birefringence of these fibres varies from one swelling agent to another in much the same way as for the wet-spun fibres, the curve, however, being displaced in the negative direction (the differences between acetylated cotton and acetylated viscose rayon are very small). The variation in birefringence is attributed to variation in its adsorption component, a concept introduced by Vermaas (*Z. physikal. Chem.*, **B52**, 131 (1942)) to explain variations in the birefringence of swollen cellulose nitrate fibres. The difference in position of the two groups of curves cannot be accounted for by differences in degree of swelling, for these are found to be small, the uptake of swelling agent being in fact greater for the acetylated fibres; it is possible, however, that in the acetylated fibres a smaller amount of swelling agent penetrates into the more highly oriented regions. A. E. S.

Properties of Man-made Fibres. P.-A. Koch. *Melliand Textilber.*, **34**, 707–710 (Aug.), 820–822 (Sept.). 914–920 (Oct.), 1025–1027 (Nov.), 1115–1117 (Dec. 1953).

First published in *Textil-Rund.*, **7**, 559–585 (Dec. 1952): cf. J.S.D.C., **69**, 172 (May 1953). H. E. N.

Chemical Processes involved in the Preparation of Synthetic Fibres. E. Müller. *Melliand Textilber.*, **34**, 850–852 (Sept.), 951–953 (Oct.), 1065–1067 (Nov. 1953).

A general review of principles, with 48 references. H. E. N.

The Textile Trade and the Newer Fibres. F. H. Clayton. J.S.D.C., **70**, 7–13 (Jan. 1954).

The three main phases of growth of the textile industry are considered. The world consumption of all fibres is still increasing, but the use of man-made fibres is increasing much more rapidly than that of the natural fibres. The properties of the synthetic fibres are summarised and some end-uses for which they are suitable. When we know what properties are required for satisfactory service for all the various end-uses, then by skilful fabric design we shall be able to make even better textile articles than we have been used to so far. AUTHOR

Attack by Moths on Synthetic Fibres in Loose Mixtures and Mixture Textiles. S. Wolf. *Faserforsch. und Textiltech.*, **5**, 68–77 (Feb. 1954).

Following his investigation of attack by moths on individual types of non-wool fibres (see J.S.D.C., **70**, 165 (April 1954)), the author examines the attack on loose fibre mixtures and shows that the various fibres can be placed in a definite order of susceptibility to attack (wool and animalised cuprammonium rayon > cuprammonium rayon, cellulose acetate, and viscose rayon > polyvinyl chloride > polyamide and polyacrylonitrile > polyester), thus enabling the behaviour of the larvæ towards any loose fibre mixture to be predicted. It is considered that the order in which the synthetic fibres fall is related to their mechanical properties, particularly shear strength, which determine their resistance to the action of the jaws of the larva. In blended yarns, particularly when highly twisted, these differences are greatly reduced, and the attack on the two kinds of fibre present tends to become equal. A. E. S.

Electrical Potential of Nylon Fibres in Aqueous Media. F. T. Wall and P. M. Saxton. *J. Physical Chem.*, **58**, 83–86 (1954): *Chem. Abs.*, **48**, 3758 (10 April 1954).

The electric potential of a nylon fibre interacting with solutions of bases is calculated from theoretical considerations and experimental data. The number of end groups on the fibre necessary to produce the fibre potential of nylon of approx. − 0·3 v. is negligibly small compared with the number of end groups that interact with the basic solutions. Fibre potential is relatively insensitive to variations in temperature. Calculations confirm the assumption that equivalent numbers of cations and anions are absorbed by the fibre. C. O. C.

Absorption of Bivalent Bases on Nylon Fibres. F. T. Wall and P. M. Saxton. *J. Physical Chem.*, **58**, 86–90 (1954): *Chem. Abs.*, **48**, 3758 (10 April 1954).

Absorption of $Sr(OH)_2$ on nylon fibres was investigated over the range $0 - 45 \times 10^{-4}$ mole. of base per g. of fibre. The results suggest that the bivalent cations are absorbed by the carboxyl ions in predominantly two forms $COOSr^+$ and $COOSrOH$. Equilibrium constants were calculated. C. O. C.

Amide-linked Derivatives of 6-Aminocaproic Acid. H. West. *Faserforsch. und Textiltech.*, **5**, 145–155 (April 1954).

By a series of reactions reminiscent of the Fischer synthesis of polypeptides substances of formula—

$$NH_2{\cdot}[CH_2]_5{\cdot}CO{\cdot}[NH{\cdot}[CH_2]_5{\cdot}CO]_n{\cdot}OH$$

(n = 1, 2, 3, 4, and 5) are prepared and characterised. Their use as model substances in investigations on nylon 6 is envisaged. A. E. S.

Structure of Macromolecular Substances. VI— Rate of Sorption of Phenols by Polyamide Fibres. A. B. Pakshver and E. K. Mankash. *J. Appl. Chem. U.S.S.R.*, **27**, 182–188 (Feb. 1954).

Kinetic and equilibrium measurements are made for the absorption of phenol, resorcinol, salicylic acid, tannin, urea, and ε-caprolactam by drawn and undrawn polyamide fibres. The diffusion coefficients (D) are in the order— phenol > resorcinol > salicylic acid > urea ≪

tannin and ε-caprolactam. They are greater for the undrawn fibre, and they increase with increase of concn. at a greater rate for undrawn fibre than for drawn fibre. The equilibrium absorption of phenol is a linear function of the concn. of the soln. and is higher for undrawn fibre. The changes in fibre length during sorption cannot, as previously stated (see *J.S.D.C.*, **70**, 31 (Jan. 1954)), be treated as a process that proceeds precisely parallel to the diffusion process— changes in length do not begin until a definite interval has elapsed after the beginning of the diffusion process (saturation of some amide linkages without appreciable disturbance of the arrangement of chain molecules), and then, although the changes accord with the diffusion equation (*loc. cit.*), this equation yields a value of D which is higher than that for the actual diffusion process. A. E. S.

Effects of Irradiation on Nylon and Polyethylene Terephthalate. K. Little. *Nature*, **173**, 680 (10 April 1954).

When specimens of oriented 66 nylon are irradiated in the Harwell pile with a dose 50 times as heavy as that which first affects the solubility, it is found that gaseous H_2 is evolved at a rate which decreases as the radiation dose increases. Groups of atoms are broken off to form low-mol.wt. compounds which can be extracted. X-Ray examination suggests that the crystallites are unaffected; a dose can, however, be applied which is sufficiently heavy to disrupt the crystallites and cause complete loss of strength. Swelling in formic acid leads to disappearance of the crystallites, and subsequent washing and drying gives new unoriented crystallites, which have lower crystallinity and a diffraction ring spacing consistent with their being methyl-substituted nylon. This unoriented nylon can be oriented by stretching by an amount similar to that in drawing ordinary undrawn nylon. When irradiated nylon, already highly swollen in formic acid, is put into formic acid plus a few % of HCl, immediate solution takes place, although conditions are such that neither peptide nor $-CH_2{\cdot}CH_2-$ bonds would be broken. Other properties, as shown by reprecipitation and examination of films, are as expected from an alkyl-substituted polyamide. Irradiation in presence of air leads to rapid loss of strength with an increase in carboxyl end-groups. Terylene behaves very differently. Hydrogen and other gases are not appreciably evolved. After a smaller dose than that used on nylon the fibres begin to powder, with complete strength loss, and polymer chips become brittle. Crystallinity does not change; irradiated non-crystalline chips remain non-crystalline, but are transformed to the normal well ordered state by annealing. Bridge formation has not, therefore, occurred, nor have side-groups appeared. Results are, in fact, consistent with straightforward chain breakdown. In absence of air there is a large increase in –COOH end-groups, whereas in presence of a plentiful supply of air the extra groups are –OH and $>C=O$, which suggests that chain breakage is in a different place in the two cases. J. W. B.

Acrylic Fibres. II— Polymerisation of Acrylonitrile. A. Hunyar and H. Reichert. *Faserforsch. und Textiltech.*, **5**, 1–8 (Jan. 1954).

Available methods for the polymerisation of acrylonitrile are discussed, particularly with respect to the preparation of polymer suitable for fibre production, for which the mean value of, and degree of variation in, mol.wt. are of prime importance. Experiments are described in which the effects of the concn. of redox initiator system ($K_2S_2O_8 + K_2S_2O_5 + FeSO_4.(NH_4)_2SO_4$), degree of agitation, and temp. on the course of the polymerisation of acrylonitrile in aq. soln. (soln.–suspension polymerisation) are determined, and it is shown that it is possible to carry out polymerisation of the type desired without stirring or cooling, the effect of spontaneous rise in temp. (which by itself would activate the redox system and lead to fractions of lower mol.wt.) being compensated by the effect of the fall occurring in the concn. of the redox system as reaction proceeds (which by itself would lead to fractions of higher mol.wt.). The possibility is examined of extending the procedure to other methods of polymerisation, to other monomers, and to copolymerisation. A. E. S.

PATENTS

Continuous Spinning of Viscose Rayon. Courtaulds. *BP* 707,141

A simplified process for the continuous spinning of viscose rayon thread consists in regenerating the freshly coagulated thread containing $> 5\%$ acid in an atmosphere of 90–98% R.H. at 30–45°C., washing, lubricating, and drying the advancing thread. W. G. C.

Skin Disintegrator for Fibre Production. Anstalt Unda. *BP* 708,626

Waste animal skin material is disintegrated sufficiently thoroughly, yet without undue fibre breakage, for production of artificial fibres or films of good strength. This is effected by a rotatable unit having a series of annular plates or discs arranged in a spaced relationship along the axis, the outer peripheries having teeth which form a working surface for engaging and transfixing the skin. An opposing rotor, with similar discs in spaced relationship to those on the first, rotates in an opposite direction and thus shreds the skin. J. W. B.

Insolubilising Regenerated Protein Fibres. ICI. *BP* 708,057

Use of sodium sulphate as the main saline constituent in the acidified saline coagulating bath, in the relaxing solution, in the formaldehyde solution at pH 4–7 and in the formaldehyde solution strongly acidified with H_2SO_4, when forming filaments from solutions of proteins, render economies possible especially in the strength of the liquors used and enables the filaments to be formed in absence of tension. C. O. C.

Dimethyl Phosphite as Low Boiling Solvent for Copolymers of Acrylonitrile. Chemstrand Corpn. *USP* 2,641,585

Dimethyl phosphite is an excellent solvent for copolymers of $\leqslant 70\%$ of acrylonitrile and $\geqslant 30\%$ of other polymerisable monomers. C. O. C.

Imparting Affinity for Dyes to Acrylonitrile–α-Halogenoacetate Copolymers. Chemstrand Corpn. *USP* 2,643,992

Copolymers of $> 80\%$ acrylonitrile and $< 20\%$ of an unsaturated ester of an α-halogenoacetic ester when treated with tris(dimethylamido)-phosphite are readily dyed deeply with acid dyes. Preferably the phosphite is added to the spinning solution before the fibres are formed. C. O. C.

Stabilised Solutions of Polyacrylonitrile. Eastman Kodak Co. *USP* 2,642,408

Addition of certain organic or inorganic acids, e.g. acetic, oxalic, phosphoric or phosphorous acid, to polyacrylonitrile dissolved in *NN*-dimethylacetamide, stabilises the solvent against decomposition caused by polyacrylonitrile acting as a catalyst. Such a stabilised solution can be used to produce white, lustrous fibres. C. O. C.

Glass Composition for forming into Fibres. Owens-Corning-Fiberglas Corpn. *USP* 2,640,784

Resin-coated Glass Filaments. Bataafsche Petroleum. *BP* 706,832

Application of glycidyl ether of a polyhydric phenol or a polyhydric alcohol to glass filaments immediately after they are formed and hardening this coating before the filaments are gathered into a strand imparts a hard coating to the filaments which is not removed by solvents whereas if the coating is applied to the filaments some time after their formation it is readily removed by solvents. C. O. C.

Static Electricity and its Removal (I p. 303).

Basic Linear Polyureas and Polyurethanes (III p. 304).

Solvents for Polyacrylonitrile (III p. 305).

Effect on Wool of Boiling in Aqueous Solutions. I— Solutions at pH 1·5–9 with and without Sodium Sulphate (VIII p. 319).

Characterisation of Occupational Hazards arising from the Presence of Carbon Disulphide (XIV p. 330).

VII— DESIZING; SCOURING; CARBONISING; BLEACHING

Comparative Detergency of Several Built Polyethenoxy Alkanoates. A. T. Ballun, J. N. Schumacher, G. E. Kapella, and J. V. Karabinos. *J. Amer. Oil Chem. Soc.*, **31**, 20–23 (Jan. 1954).

Polyethenoxy alkanoates of varying ethenoxy chain length were prepared, and comparative detergency values were obtained. It was noted that approx. two-thirds of one ethenoxy group for each carbon atom in the hydrophobic portion of the molecule was essential for optimum detergency values. The non-ionic esters prepared from lauric and myristic acids possessed substantially greater detergency than pelargonic and stearic acids. When 12-hydroxystearic acid was likewise condensed with ethylene oxide, no ethenoxy groups were found attached to the secondary hydroxyl group. A partial explanation for this behaviour, based on the addition of ethylene oxide to the hydroxyl group of greatest acidity, is given.

P. G. M.

PATENTS

Bleaching Proteinaceous Substances. Tennants Consolidated. *BP* 708,561

All proteinaceous substances are well bleached without any other property being modified by treating them with a chlorite and a peroxide or persalt and then with a reducing agent. Bleaching is preferably done in three steps as follows: (1) treat with a persalt of high oxidation potential, e.g. a persulphate, (2) then treated with an alkali or alkaline earth chlorite, and (3) treated with a bisulphite, hydrosulphite, formaldehyde or urea. C. O. C.

Stabilised Bleached Polyacrylonitrile Fibres. DuP. *USP* 2,643,934

White polyacrylonitrile fibres the colour of which is stable to ultraviolet radiation are obtained by treating the raw polyacrylonitrile fibres first with an aqueous solution containing active chlorine and then with hypophosphorous acid or an alkyl or aryl phosphite and finally heating. C. O. C.

Laundering. Seifen-und Glyzerinfabrik C. Hartung. *BP* 707,908

The goods are treated in a hot aqueous solution of a surface active, oxygen-yielding material, there being enough of the latter to give the same oxygen yield as is given by 10 g. of Na perborate for each 10 litres of water, as a stabiliser. Suitable stabilisers are sodium pyrophosphate, hexametaphosphate, tripolyphosphate or tetrapolyphosphate or at high pH $MgSiO_3$. The goods are kept in the solution until clean, the solution being allowed to cool the whole time. C. O. C.

Surface-active Properties of Salts of α-Sulphonated Acids and Esters (III p. 304).
Pressure Dyeing and Bleaching with special reference to Recent Developments (VIII this page).
Carpet Soiling (XIV p. 330).

VIII— DYEING

Dyeing above 100°C. W. Justus. *Melliand Textilber.*, **34**, 1148–1151 (Dec. 1953).

Above 100°C. the affinity of direct cotton dyes decreases, but levelling increases. The swelling of rayon decreases above 100°C., and equally well penetrated pack dyeings can be obtained at 115–120°C. in 1–2 hr. as at 90°C. in 6 hr., the former leaving the yarn in better condition. Acid dyes dye wool 3–4 times as rapidly at 110°C. as at 98°C.: i.e. a dyeing time of 15–20 min. is sufficient at 110°C. Better levelling is achieved only in longer periods (1 hr.) at high temp. Investigation of the wet strength of fibres dyed at pH 5 for various times at various temp. shows that above 110°C. degradation suddenly becomes more serious. Dyeing at various temp. to completion gives approx. the same reduction in wet strength, but the wet strength progressively falls off with increasing temp. if dyeing is carried out for the same length of time at each temp. Chroming for a short time at a higher temp. is more beneficial than the normal process. Large-scale dyeing, abrasion, and bursting tests, and spinning trials, have shown wool dyed at high temp. (106°C.) not to be inferior. The wet strength of wool dyed for 90 min. at 98°C. and wool dyed for 30 min. at 108°C. is the same if the pH is that of the isoelectric point of wool, but the wool dyed at high temp. is weaker the further the pH is removed from this point: i.e. pH 3–6·5 is most suitable for high-temp. dyeing. Wool dyes are generally stable up to 110°C., but alteration in hue may occur with pale dyeings. High-temp. dyeing is important for better equalising with direct cotton dyes, shorter dyeing times with wool dyes, and greater uptake in the dyeing of synthetic fibres.

H. E. N.

Pressure Dyeing and Bleaching with special reference to Recent Developments. F. H. Marsh. J.S.D.C., **70**, 105–112 (March 1954).

Direct Cotton Dyes for Dyeing above 100°C. E. Herrmann. *Melliand Textilber.*, **34**, 759–760 (Aug.), 1072–1073 (Nov. 1953).

Further lists are given of FBy dyes suitable for application above 100°C., suitable for application above 100°C. only in the presence of protective agents (0·25–0·5 g./l. Monochrome mordant or, for Benzo Fast Copper dyes, 1 g. Sustilan N per litre), and unsuitable for this purpose. The ranges examined are— Benzo, Benzo Viscose, Benzo Fast Copper, and Benzamine dyes. H. E. N.

Metal-complex Direct Cotton Dyes. R. Paltzer. *Melliand Textilber.*, **34**, 1151–1154 (Dec. 1953).

Dyeing Coprantine (Ciba) dyes from a bath containing 0·5–2·5% calcined tetrasodium pyrophosphate instead of soda gives the following advantages— lower pH, hence less swelling; better dispersion of dyes; complexing of heavy metal ions; improved reservation of cellulose acetate rayon. Trilon must not be added to baths containing metal-complex dyes, as it may remove the metal from them. H. E. N.

Effect of Auxiliary Products on Dyeing with Direct Cotton Dyes. M. Haas. *Melliand Textilber.*, **34**, 960 (Oct. 1953).

Addition of an alkylarylsulphonate–alkyl sulphate mixture (Comprox A, Anglo-Iranian Oil Co.) increased the capillary rise of some dyes on filter paper, but in practice, although the auxiliary product improves levelness, no improvement in the penetration of dyeings was found. H. E. N.

Adsorption and Diffusion of Direct Cotton Dyes in Cellulose Fibres. P. W. Lange and E. Lindvall. *Svensk Papperstidning*, **57**, 235–241 (15 April 1954).

The amount of dye adsorbed at equilibrium depends on the technical treatment of the fibre and seems to be proportional to the amount of adsorbed water corresponding to a unimolecular layer in accordance with the Brünauer–Emmett–Teller (B.E.T.) equation. The adsorption process can be described by the Freundlich adsorption isotherm. The amount of dye irreversibly bound was measured for the various fibres and from measurement of the diffusion process a diffusion constant was determined for the interior of the fibres. A non-uniform distribution of matter and pores in the fibre wall was held to account for the difference in diffusion rate found for the outer and inner regions of the fibres.

S. V. S.

Azoic and other Fast Dyeings on Knitted Cotton Fabric. E. R. Wiltshire. J.S.D.C., **70**, 41–46 (Feb. 1954).

Methods of dyeing the azoic dyes on knitted cotton fabrics on the winch are described. The application of the coupling component is straightforward, but development can cause difficulty. Relatively few developers can be processed satisfactorily on the winch, and as a general method pad development is recommended. For this purpose the fabric should preferably be dried after application of the coupling component. The final after-treatment of azoic dyeings is done very efficiently on the winch. Pale shades can be dyed with vat dyes on the winch if adequate quantities of Dispersol VL are employed. The technique described is simple and has advantages over the use of solubilised vat dyes, particularly for blues.

AUTHOR

"Naphthanil" Coupling Components— Relationship between Chemical Structure and Substantivity for Cellulose. L. Spiegler. *J. Org. Chem.*, **18**, 1292–1316 (1953): *Chem. Abs.*, **48**, 2377 (25 Feb. 1954).

Curves showing the absorption by cellulose of a large number of arylamides are given and relation to their

structure. Those having a linear configuration are more substantive than angular types; those which are flat or coplanar have better affinity for cotton than analogous or isomeric noncoplanar compounds; substitutions or modifications which disrupt or disturb the coplanar configuration of the parent substance nucleus result in lessened substantivity; maximum substantivity occurs with compounds which are predominantly linear and coplanar in configuration and which contain several substantive groups which are 10A, or multiples thereof apart, corresponding to the periodicity of the cellulose units forming the cellulose chain. The relationships are similar to those observed for direct dyes and agree with the concept that co-ordinated H supplies the link between cellulose and the direct dye or the coupling component.
C. O. C.

Azoic Dyeing of Regenerated Cellulosic Fibres in Package Form. W. Kirst and K. Gehre. *Melliand Textilber.*, **34**, 857–858 (Sept.), 960–963 (Oct. 1953).

Regenerated cellulose absorbs alkali more readily than cotton. In spite of the reduced quantities of NaOH used, more is present on the fibre, particularly in the presence of much salt, and requires more alkali-binding agent in the development liquor. To reduce swelling, the excess NaOH normally employed can be replaced by calcined soda (3–4 g./litre) and a protective colloid (Lamepon A, Chem. Fabrik Grünau AG) at 45°c. Exhaustion is obtained by means of salt. Suitable selections of bases and Naphtols are given. Ofna-lan (NCO) dyes sodium Naphtolates marketed for application on wool–rayon staple yarn, are suitable for this soda process.
H. E. N.

"Naphthol" Dyeing. R. H. Nuttall. *Amer. Dyestuff Rep.*, **43**, P178–P179 (15 March 1954).

Methods for applying azoic coupling components to cotton cloth, yarn, and raw stock are given, and mention is made of some causes of uneven dyeing and dyeings of poor fastness to rubbing. A recipe for dyeing polyethylene terephthalate with azoic coupling components is provided.
J. W. B.

Dyeing, Printing, and Chemical Finishing—Recent Developments. F. Weber. *Melliand Textilber.*, **34**, 1079–1080 (Nov.); 1161–1165 (Dec. 1953).

A general review, with references. H. E. N.

Effect on Wool of Boiling in Aqueous Solutions. I—Solutions at pH 1·5–9 with and without Sodium Sulphate. R. V. Peryman. J.S.D.C., **70**, 83–92 (Feb. 1954).

Wool of 64s quality, in the form of yarn and cloth, has been given boiling treatments of 3 hr. with 50 min. for raising to the boil to imitate prolonged commercial dyeing processes but without the addition of dye. The pH of the solutions has been controlled at selected pH values between 1·5 and 9 by the gradual addition of sulphuric acid or sodium carbonate during the boiling. The wool has subsequently been examined for modification by means of determinations of alkali solubility, soluble nitrogen, disulphide sulphur, total sulphur, breaking load air-dry and wetted with water, extension at break, abrasion resistance, and loss in weight. A smaller quantity of 56s Corriedale wool has been treated concurrently though subjected to fewer tests. The conclusions reached are—(1) the wools have suffered minimum modification when boiled at pH 3–3·5; (2) sodium sulphate, in concentrations up to 9·2 g./litre, has shown relatively little effect on the wool within the pH range 1·7–6·8, but above pH 6·8 the presence of sodium sulphate has caused alkali damage, which increases rapidly with increase of salt concentration and rise of pH; (3) a strong correlation has been found between disulphide content and wet breaking load for treatments having pH values above 4; a similar correlation has been found to apply to abrasion resistance after about 20% of the original disulphide sulphur has been broken down; (4) alkali solubility and soluble nitrogen have shown a strong correlation with abrasion resistance and to a lesser extent with wet strength for treatments having pH values below 2·5. The practical implications are discussed.
AUTHOR

Effect of Dyeing on the Sorption of Water Vapour by Wool. P. Larose. J.S.D.C., **70**, 77–81 (Feb. 1954).

The reduction in the sorptive capacity of wool for water vapour due to acid treatment or dyeing of the wool is discussed and previous results are reviewed. Preliminary experiments carried out with a view to showing a relation between the reduction in the amount of water sorbed and the amount of dye in the wool are reported. The results of these experiments point to an equivalence between the dye in the wool and the reduction in regain of the dyed wool as compared with undyed wool. AUTHOR

Alteration in the Mechanical Properties of Wool caused by Dyeing Processes. G. Mazingue, P. Outreman, M. van Overbeke. *Bull. Inst. Textile France*, (42), 23–28 (Oct. 1953).

Samples of wool were dyed at three different pH values and the mechanical properties of the processed specimen examined. G. L.

Afterchrome Process. J. F. Gaunt. J.S.D.C., **70**, 46–57 (Feb. 1954).

The following aspects of the afterchrome process have been examined—(1) the dyeing of afterchrome dyes prior to chroming, (2) the effect of dichromates on wool during afterchroming, and (3) the effect of dichromates on chrome dyes during afterchroming. The main difference between chrome dyes and acid dyes of comparable constitution is the greater sensitivity of the former to precipitation by acid. Several of the more commonly used chrome dyes are out of solution under normal dyeing conditions, and yet have excellent level-dyeing properties. The rate of uptake of chromium by wool during afterchroming is independent of the presence of dye on the wool. When afterchroming at the boil, the pH of the liquor rises steadily, owing to the reduction of chromic acid to neutral tervalent chromium compounds, but this effect is much less marked at 80° and 90°c. The rate of absorption of chromium is very sensitive to the pH of the chroming bath, but is only slightly affected by temperature. During afterchroming at low pH values, there is a tendency for certain dyes to be destroyed by oxidation. The chrome lakes formed by afterchroming with dichromates have much better wet fastness than those formed by after-treatment with salts of tervalent chromium.
AUTHOR

Afterchrome Process. C. M. Whittaker. J.S.D.C., **70**, 188 (May 1954).

Substratum Form in Wool Dyeing. A. K. Setty. *Amer. Dyestuff Rep.*, **43**, 98–100 (15 Feb. 1954).

Studies on the rate of wool dyeing are carried out on wool in fabric, yarn, and loose-fibre form, using Alizarine Sapphire BN (*C.I.*1054). To eliminate variables due to processing damage, the yarns are obtained by dissecting the fabric and the loose fibres by teazing out the yarns. It is concluded that the equilibrium exhaustion of the dyebath is not appreciably affected by the physical form of the substratum, but the time of half dyeing decreases in the order fabric > yarn > fibre, the first decrease being greater than the second. Similarly, the overall rate of dyeing depends on the form of substratum. The initial dyeing rate is most affected. The middle straight portion of the curve relating proportion of total exhaustion at any time with the square root of dyeing time represents the true dyeing characteristic of a dye for the wool fibre. The initial part represents the time required for the dye to reach the fibre, and the part beyond the straight portion represents the dyeing to exhaustion that takes place after the dye has reached the fibre centre. The main conclusion is that all fundamental studies of dyeing rates should be carried out with loose fibres rather than with yarn or fabric. J. W. B.

Dyeing Wool Blends and other Fibres for Carpets. H. F. Clapham. *Amer. Dyestuff Rep.*, **43**, P141–P142 (1 March 1954).

Neutral Premetallised Dyes. C. Weidmann. *Amer. Dyestuff Rep.*, **43**, P167–P171 (15 March 1954).

Structures of acid- and neutral-dyeing premetallised dyes, and of chromium–dye complexes formed in chrome dyeing, are briefly compared, though no mention is made of the groupings which confer water-solubility on the neutral-dyeing types. In discussing dyeing methods emphasis is

placed on pH control, and techniques are described for attaining it. As dyeing takes place near the neutral point, it is essential to have a suitable water supply and to avoid trouble due to alkalinity in scoured wool and acidity in carbonised wool, the latter being particularly difficult to neutralise adequately. J. W. B.

New Neutral-dyeing Metallised Wool Dyes. H. F. Clapham. *Amer. Dyestuff Rep.*, **43**, P200–P202, P208 (29 March 1954).

New neutral-dyeing metallised wool dyes may replace chrome-dyeing types for light and medium depths, as they are easy to apply and are of excellent fastness. Fastness to carbonising, decatising, and chlorination is also good. Stripping to a pale colour is simple, and certain members are equally useful for nylon. J. W. B.

Recent Researches into the Dyeing Properties of Vat Dyes. J. Wegmann. *Teintex*, **19**, 83–99 (Feb. 1954).

The effects of variations in caustic soda concn., time and temp. of dyeing, and salt concn. on the resultant depth of dyeing obtained on both cotton and viscose rayon have been studied with representative Cibanone (anthraquinone vat) dyes, and are presented in graphical form. B. K.

Electrolytic Vatting of Dyes. G. Dierkes and W. Wansleben. *Melliand Textilber.*, **34**, 958–959 (Oct. 1953).

Using a spongy nickel cathode, a carbon anode, a current of 3 amp. at 18 v., and a caustic soda soln. containing a little wetting agent, the following Indanthren dyes were vatted in 6–20 min.: Yellow G, Golden Yellow GK and RK, Orange F3R and GK, Brilliant Orange GR, Scarlet R, Brilliant Violet RR, RK, and BBK, Brilliant Green GG, and Olive 3G. Others required very much longer or would not vat. Dyeings produced by means of electrolytic vatting were equal to those effected normally. H. E. N.

Effects of Soaping on Vat Dyeings. H. H. Sumner and T. Vickerstaff. J.S.D.C., **70**, 123 (March 1954).

Dyeing with Disperse Dyes. A. E. Stubbs. J.S.D.C., 120–122 (March 1954).

Dyeing of Acetate Rayon with Disperse Dyes. I— Aqueous Solubility and the Influence of Dispersing Agents. II— Relation between Aqueous Solubility and Dyeing Properties. C. L. Bird. J.S.D.C., **70**, 68–77 (Feb. 1954).

Aqueous solubilities have been determined at 25° and 80°C. for a range of purified disperse dyes. The solubilising effect of a number of dispersing agents has been examined. It is shown that aqueous solubility has some bearing on the rate of dyeing of disperse dyes and largely governs their levelling power. Addition of a dispersing agent to an acetate rayon dyebath reduces the percentage exhaustion to an extent which is proportional to the degree of solubilisation. AUTHOR

Dyeing of Nylon Yarn in Package Form. W. S. Willson. J.S.D.C., **70**, 22–24 (Jan. 1954).

A method of dyeing continuous-filament nylon yarn in cake form has been developed and proved on a bulk scale. The "mock" cakes are wound on a converted coning machine, and dyeing is carried out on standard package-dyeing machinery. The use of various classes of dye is discussed, and suitable technique and aftertreatment are described. AUTHOR

Dyeing of Synthetic Fibre Blends. L. S. Thompson. *Canadian Textile J.*, **71**, 53–57 (26 March 1954).

This review includes methods of dyeing the following blends— viscose rayon with acetate rayon, nylon, Vicara, Dacron, dynel, Orlon 42, Acrilan, and wool; wool with Orlon, Vicara, Dacron, Acrilan, nylon, and dynel; and viscose rayon–acetate rayon–wool. J. W. B.

Dyeing and Finishing Piece Goods made from Dynel–Rayon and Dynel–Rayon–Acetate Blends. F. C. Rippner. *Amer. Dyestuff Rep.*, **43**, P209–P212 (29 March 1954).

Problems of the Garment Dyer and Finisher, including the Dyeing of Buttons. B. Kramrisch and A. E. Bratt. J.S.D.C., **70**, 173–183 (May 1954).

Diffusion of Dyes in Gelatin. E. E. Jelley and R. B. Pontius. *J. Phot. Sci.*, **2**, 12–25 (1954): *Chem. Abs.*, **48**, 3824 (10 April 1954).

Diffusion of a dye through or from a gelatin layer on a film base is arrested instantly by plunging the open-funnel into a solid CO_2–alcohol bath at — 80°C. so as to freeze the water present. It is then transferred to a drying chamber in which the ice is volatilised from the gelatin in a current of cold dry N. The dried sample is cross-sectioned with a microtome and mounted, after which it is scanned with a recording high-resolution microdensitometer of new design. Densitometric curves obtained in this way a simple yellow azo dye and with four magenta 3-azobenzanthrone dyes are given and discussed. The concentration profile of a diffused dye can change at different values of dyebath pH and presence of a mordant can alter the concentration profile. The diffusivity and substantivity of a dye can be greatly altered by certain relatively minor changes in dye structure. The process of transfer of dye by diffusion from a matrix into a mordant blank is illustrated and the rôle of the mordant discussed. C. O. C.

PATENTS

Dyeing Cellulose Fibres with Copper or Nickel Complexes of Direct Azo Dyes. Ciba. *BP* 708,114

Very satisfactory dyeing of cellulose with copper or nickel complexes of direct azo dyes is obtained if there is present in the dyebath an aliphatic amine containing at least two N atoms each bound to two C atoms and at least two OH groups, 1:2-di-(β-oxyethylamino)-ethane being particularly suitable. C. O. C.

Dyeing in a Molten Metal Machine with Dyes Susceptible to Permanent Reduction. Standfast Dyers and Printers. *BP* 707,462

When applying dyes which are susceptible to permanent reduction, e.g. Alizarin Fast Green GGW (CAC), Carbolan Brilliant Green 5GS (ICI), Polar Brilliant Blue RAW (Gy) or Duranol Brilliant Blue G (ICI), any tendency to reduction is avoided by treating the material with a mild oxidising agent, preferably added to the dye liquor, before it enters the molten metal. Suitable oxidising agents are sodium *n*-nitrobenzene sulphonate, sodium nitrotoluene-*p*-sulphonate, sodium ortho vanadate, sodium chlorate and sodium nitrite. C. O. C.

Dyeing of Animal Fibres with Vat or Sulphur Dyes in a Molten Metal Machine. Standfast Dyers & Printers. *BP* 708,934

Animal fibres are dyed with vat or sulphur dyes in a molten metal machine by adding to the dyebath an organic ester which hydrolyses in presence of the dye liquor at the temperature of the metal bath to liberate acid which neutralises the alkali in the dye liquor. The ester should be such that it hydrolyses slowly but completely during the passage of the goods through the metal. Alternatively the fibres may be impregnated with the ester before entering the dyebath. Suitable esters are ethyl lactate, *n* or *iso*propyl acetate, and diethyl tartrate. C. O. C.

Levelling Agents for Wool Dyes containing No or only One Sulphonic Acid Group. Forbe Dyeworks. *BP* 707,925

The water-soluble condensate of urea or HCHO with an aromatic sulphonic acid containing one or more phenolic OH groups are useful levelling agents for use with wool dyes containing no or only one sulphonic acid group. At least 0·75% on the wt. of the goods or 0·0025% on the wt. of the liquor should be used. C. O. C.

Azoic Dyeing of Acetate Rayon. Celanese Corpn. of America. *BP* 706,735

Improved dyeings are produced on acetate rayon with azoic dyes by treating with a swelling agent between diazotisation and coupling, e.g. it is possible to get the same colour value on acetate rayon with 4-benzoylamino-2:5-diethoxyaniline-2:3-hydroxynaphthoic anilide as is obtained on cotton. C. O. C.

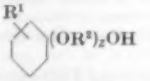
Dyeing Cellulose Esters and Synthetic Polymers with Vat Dyes. Celanese Corpn. of America. *BP* 707,010

A vat made up of a vat dye, a reducing agent, an alkali metal carbonate, an alkylolamine, e.g. mono- di- or triethanol or propanolamine, and a liquid swelling agent, e.g. monoethyl ether of diethylene glycol or triethyl

phosphate. Particularly good results are obtained when for each 100 parts by weight of water there are used 0·1–1·0 of carbonate, 0·5–10·0 of alkylolamine and 3–25 of the swelling agent. C. O. C.

Hair Colour-rinse Concentrate. Rayette. *USP* 2,643,211

An aqueous mixture of a water-soluble dye and a compound of formula—



(R^1 = Alk of 1–12C and is preferably of the branded type; R^2 a lower alkaline group, z = 1–5); the two substituents may be *o*, *m* or *p*. The dye chosen should be less soluble in the above compound than it is in water so that on application to the hair it only dissolves slowly therein and penetrates there through into the hair. C. O. C.

Azoic Dyeings on Leather (IV p. 311).

Heat Setting of Terylene Polyester Filament Fabrics in relation to Dyeing and Finishing (X p. 322).

Selective Adsorption of Dyes by Synthetic Resins (XIII p. 326).

IX— PRINTING

Printing of Terylene Polyester Fibre. A. G. H. Michie. *J.S.D.C.*, **70**, 13–15 (Jan. 1954).

Experimental work has shown that the Dispersol and Duranol dyes, Alcian Blue 8G, pigments in the form of pigment printing compositions, and vat dyes may be applied to Terylene fibre by printing techniques. The methods of application are outlined with stress being placed on the use of pressure steaming and swelling agents for the disperse dyes. Vat dyes are applied by the acid-leuco technique and require to be steamed at high pressures for satisfactory fixation. Their light fastness when printed on Terylene fibre is lower in practically all cases compared with prints on cotton. The mechanical conditions necessary to obtain prints of satisfactory quality on Terylene fibre are discussed. AUTHOR

PATENTS

Printing Assistants. ICI. *BP* 708,248

Sulpholanes, e.g. sulpholane or dimethylsulpholane (2:4-dimethyltetrahydrothiophene dioxide) are excellent printing assistants for use on all types of fibres and with most dyes or dye components, thus added to a printing paste containing Caledon Blue RCS (*C.I.*1114) much stronger and brighter prints are obtained than in absence of a sulpholane. C. O. C.

Printing with Diazo Compounds. Kalle & Co. *BP* 708,834

Compounds of formula—

(X = SO_2·OAr, COOAr, SO_2·NY·Ar or CO·NH·Ar; (Y = H or an aliphatic or aromatic residue)), e.g. benzoquinone —(1:2)–diazide–(2)–4–(*N*:*N*-diphenyl)–sulphonamide, are used as the light sensitive component which after being acted upon by light can be removed by alkali. C. O. C.

Printing with Suspension of Organic Pigments in Aqueous Starch. DuP. *USP* 2,641,554

The printing paste consists of aqueous starch, an organic pigment, a binder and an acid catalyst. The binder consists of a water-soluble triazone resin obtained by condensing at pH 0·25–4·0 urea, formaldehyde, and a linear polyamine of 2–8 C and containing 2 primary amino groups zero — three secondary amino groups, each amino group being separated from the others by 2 C atoms; it contains at least 1·5 triazone rings and at least 3 methylol groups attached to N atoms per mol. of linear polyamine. After printing and either drum drying or steam- and acid-ageing prints of good fastness to washing, rubbing, chlorine and light are obtained. The process is cheaper than most resin-pigment printing processes and there is no tendering of the fabric. C. O. C.

Metal Coatings on Plastics. J. Garofano. *USP* 2,642,390

Complete adhesion either in large patches or very fine lines is obtained between a metal coating and a plastic by first treating the plastic with a cellulose lacquer (this step is unnecessary with cellulosic plastics). The plastic is then covered with a thin sheet of paper on which has been printed the negative of the design it is desired to apply, this negative being printed with an ink made up of rosin, wax, an oil and a dye, e.g. carbon black. The paper is then removed leaving the negative design on the plastic which is now treated with a suspension of metal in acetone by means of a spray gun. The resist is then removed with a solvent and the metal deposit polished or burnished. If desired a further layer of metal may be deposited electrolytically into that already present. C. O. C.

Colour Photography. Eastman Kodak Co. *USP* 2,640,776

Stable combinations of silver halide emulsions, sensitising dyes and colour couplers are described which can be melt-coated without loss in sensitivity. C. O. C.

Diazotype Printing. Chemische Fabriek L. van der Grinten. *BP* 708,151

A method of producing diazotype copies by selective exposure (i.e. exposure to light of locally different intensities) of single-component diazo layers sensitised with a *p*-aminobenzene diazo compound.

BP 708,204

Addition of an alkali orthophosphate to diazotype developers containing an acrylacetanilide in which the acryl radical is $H(CH_2)_nCO$(n = 1–3) enables use of concentrated developers which yield well developed prints using thin-film development.

BP 709,359

Modification of *BP* 708,151 (above). Tonal reproduction is controlled by using in addition to the *p*-aminobenzene diazo compound a second diazo compound which yields a photochemical decomposition product which is less active than that derived from the *p*-aminobenzene diazo compound. C. O. C.

Stable Photographic Colour Images. Agfa A-G für Photofabrikation. *BP* 706,792

The stability of photo mechanically produced multi-colour prints is much increased by treating them at any stage during or after their production or processing with substances which absorb ultraviolet radiation but which do not otherwise affect the prints, e.g. stilbene, pyrene, hydroxyquinoline, arylacetophenone, diamidobenzophenone and derivatives of these compounds and with aliphatic and/or aromatic aliphatic aldehydes and/or derivatives of such aldehydes reacting as aldehydes. C. O. C.

Coloured Photographic Positives. International Polaroid Corpn. *BP* 706,921

Photomechanical Printing Plates. Kalle & Co. *BP* 706,879

Water-insoluble azo dyes containing at least one residue of an *o*-quinone diazide and in addition to the azo group at least one auxochromic group derived from a sulphonic or carboxylic acid of the diazide, the azo dye portion of the molecule being coupled in an ester- or amide-like manner to the acid group, are useful as light sensitive dyes in the photomechanical preparation of printing plates. On exposure to light the benzene ring containing the diazo group is split yielding the dye carboxylic acid which is readily separated from the diazo compound by alkaline liquor. This yields an orange to deep violet positive image which readily takes up greasy ink. C. O. C.

Fluorescent Brightening Agents for improving Whites of Photographic Prints (IV p. 313).

Dyeing, Printing, and Chemical Finishing— Recent Developments (VIII p. 319).

Diffusion of Dyes in Gelatin (VIII p. 320).

X—SIZING AND FINISHING

Occurrence and Prevention of Biological Attack of Cellulosic Fibres. R. G. Fargher. *Biochem. Soc. Symposia* (Cambridge), (11), 71–84 (1953): *Chem. Abs.*, **48**, 4224 (10 April 1954).

Review of present knowledge of the mechanism of attack by micro organisms on cotton and bast fibres of methods of rotproofing. C. O. C.

Nature of Modifications of Wool by Chlorination, and the Effect of Shrink resisting on Wear. D. Frishman and M. Harris. *Amer. Dyestuff Rep.*, **43**, P174–P177 (15 March 1954).

Chlorination treatments are briefly surveyed, and it is shown that they vary considerably with respect to the degree of shrink resistance and the extent of wool modification. A graph, which may be likened to a phase diagram, is shown which relates the finish, in terms of colour and shrink resistance, to the amount of chlorine applied and the time taken to apply it, the operative pH being 8·4. Treatments which are rapid and thus confined to the fibre surface are better in all respects. Results are given of a test in which large-scale footwear-wearing trials are related to the alkali solubility. J. W. B.

Back-sizing Carpets and Rugs. O. B. Hager. *Amer. Dyestuff Rep.*, **43**, P172–P173, P177 (15 March 1954).

Reasons for back-sizing carpets, methods of applying sizes, and composition of sizes according to the effects desired are discussed, including the use of rubbers and synthetic resins. J. W. B.

Effect of Degree of Expression on Removal of Water from Textiles. E. Selter. *Textil-Praxis*, **9**, 178–183 (Feb.), 258–268 (March 1954).

The removal of water from textiles has been studied from the following angles—Theoretical considerations of the mechanical removal of water from textiles by squeeze rolls, pads and mangles. The relationship between expression and the following factors—(1) pressure—influence of type, diameter and composition of squeeze rolls; (2) speed of cloth; (3) period of immersion of cloth (4) type of cloth, including yarn quality and weave; (5) temperature; and (6) surface-active compounds. The main results obtained from these investigations are—Degree of expression is influenced mainly by the pressure applied and also the hardness of the squeeze rolls. Increase in speed of the cloth decreases the expression, but this varies with the material: little effect is shown with synthetic fibres, but marked effects are obtained with cotton and viscose rayon. B. K.

Crease-resisting—New Developments. H. Jörder and W. Walter. *Melliand Textilber.*, **34**, 965–969 (Oct. 1953).

To prevent migration of the precondensates to the surface of the fibres during drying, slow drying at 70–80°c. is usually recommended. This is shown to be unsound and shock-drying is preferred, particularly when carried out evenly. Condensation can also be carried out by means of a heat shock. These results were achieved using a machine (A. Monforts, München-Gladbach), which projects superheated steam (150–170°c.) at 10–12 m./sec. evenly on to both sides of the piece. The material is exposed to the high temp. only for a very short time, and the result is shown to be satisfactory by numerous tests and four photomicrographs. Steam consumption is even less than on cans. Drying and condensation can be carried out on one machine or by two in series, depending on the production required. H. E. N.

Sizing. H. Svasen. *Melliand Textilber.*, **34**, 955 (Oct). 1070–1071 (Nov. 1953).

A discussion in the light of experience. H. E. N.

Chlorination of Wool at Long Liquor Ratios. C. Earland and D. J. Raven. J.S.D.C., **70**, 185–187 (May 1954).

It is shown that the effectiveness of chlorine in acid solution as an anti-felting agent for wool is independent of the liquor : wool ratio up to 100,000 : 1. It is considered that this is further evidence for concluding that the reaction between wool and chloramines is not a simple chlorination. AUTHOR

Formation of Polymers in Wool. G. W. Madaras and J. B. Speakman. J.S.D.C., **70**, 112–116 (March 1954).

When potassium persulphate is used in place of hydrogen peroxide, it is unnecessary to impregnate wool with ferrous ammonium sulphate in order to ensure preferential polymerisation of methacrylic acid inside the fibres at 25°c. Polymerisation is initiated by the free radicals which are formed by the action of potassium persulphate on the cystine disulphide bonds. With reduced fibres, however, potassium persulphate is not much more effective than hydrogen peroxide. C. J. W. H.

Heat Setting of Terylene Polyester Filament Fabrics in relation to Dyeing and Finishing. D. N. Marvin. J.S.D.C., **70**, 16–21 (Jan. 1954).

The dry heat setting of Terylene filament fabrics is considered in relation to dimensional stability, wet creasing, stiffness and recovery from dry creasing, and dyeing properties. The effects of aqueous heat are also discussed. Setting machines are described, and the setting conditions for Terylene fabrics outlined. AUTHOR

PATENTS

Preventing Rubberised Yarns from Sticking. Lewis & Taylor. *BP* 708,278

To facilitate processing of yarns coated with rubber latex they are passed through a bath of molten hydrocarbon wax, which is solid when cool. This prevents the rubberised yarns from sticking together prior to vulcanisation, and raises no dust problems as do conventional methods for reducing stickiness. Also, belting from such vulcanised material has improved frictional properties by virtue of the permanent thin layer of paraffin wax. J. W. B.

Increasing Surface Friction of Fibres, Fabrics or Films. Deutsche Gold- und Silber-Scheideanstalt, v. Roessler. *BP* 708,269

Resistance to friction on the surface of fibres, yarns, fabrics and films (e.g. artificial leather) is induced by treatment with an aqueous suspension of finely divided SiO_2 or TiO_2, which has been obtained in the finely divided state by thermal decomposition of volatile Si or Ti compounds, e.g. halides, by oxidation or hydrolysis. The textile is loaded with $< 1\%$, and preferably 0·05–0·2%, of oxide. J. W. B.

Water-repellent Finish. Bradford Dyers' Assocn. *BP* 708,821

Fibrous materials of all kinds are rendered water-repellent by treating them with an organosilicon compound in presence of an organic or inorganic compound of titanium which can be hydrolysed or decomposed to an oxide or hydrated oxide of titanium. Thus nylon cloth treated with a solution of silicone (1·875 parts by wt.) and tetrabutyl orthotitanate (0·125) in petroleum spirit boiling at 130–180°c. (98) and dried in air at 20°c. is much more repellent than if 2 parts of either the silicone or titanate is used by itself. C. O. C.

Rendering Fibrous Materials Water-repellent. Dynamit-AG. *BP* 708,026

The materials are impregnated with a substance of formula RNXY (R = Alk of 11C; X and Y each = same or different, H or an aliphatic acid group containing >3C in the chain attached to the CO group; or X + Y = an *o*-dicarboxylic aromatic group), e.g. stearyl-heptadecylamine or heptadecylphthalimide. Tent canvases so treated are impervious to air and are rotproof. C. O. C.

Water-repellent Finish. National Lead Co. *USP* 2,641,558

Fibrous material is rendered water-repellent by treating it first with a solution containing anionic zirconium, e.g. aqueous solution of the double ammonium zirconium carbonate, containing also a fat and/or wax and then with an aqueous solution of a polyvalent metal of group III, IV, or VIII, e.g. basic zirconyl chloride, stannous chloride or ferrous sulphate. C. O. C.

Flameproofing and rendering Cellulosic Materials Water-repellent. Diamond Alkali Co. *USP* 2,640,000

The material is treated with an inorganic flame retardant substance and a chlorinated organic compound so as to keep these two substances out of contact with each other

and to keep the chlorinated compound out of contact with the cellulose while permitting intimate contact to be readily established at flame temperatures. One way of doing this is to separate the fibres from the chlorinated compound by interposing between them a film of a water-repellent substance. C. O. C.

Reducing the Felting Property of Wool. Precision Processes (Textiles). *BP* 706,626

The material is treated with an aqueous chlorite at pH 4–9 in presence of a compound of formula RCHO (R = H, CHO, COOH, subst. or unsubst. sat. or unsat. Alk or subst. or unsubst. Ar) at $\geq$100°C. so that 5–30% of the chlorite on the weight of material is used up. C. O. C.

Reducing the Felting Power of Wool. J. H. McLauchlan. *BP* 708,391

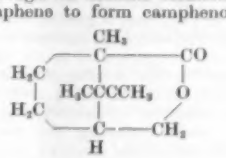
The wool is treated for 30–70 sec. with an aqueous hypochlorite at pH 2·2–3·5 and 4–24°C. while maintaining the available chlorine content of the solution at 0·035–0·09% by wt. by continuous addition of hypochlorite and acid. C. O. C.

Setting the Twist in Keratinous Yarns. Monsanto. *USP* 2,642,332

The twisted yarn is first treated with an aqueous dispersion of a reducing agent capable of splitting the cystine link, e.g. a mercapto containing at least one OH group and 2–6C, and a thermoplastic resin, e.g. a styrene-butadiene copolymer, at 100–105°F. After 1–6% on the dry weight of the keratin of the reducing agent has reacted with the keratin, the yarn is removed from the liquor and dried at 90–350°F. The treatment is preferably applied before dyeing as the treated yarn is difficult to dye satisfactorily. C. O. C.

Increasing the Strength and Durability of Textiles. British Intercontinental Corpn. *BP* 708,268

Textiles of all kinds are given increased tensile strength and resistance to wear by immersing them in an alcoholic solution of pinene hydrochloride and then treating the material with hot aqueous NaOH or Na_2CO_3 so that camphene is formed within the fibres. The goods are then passed through an acetone solution of $KMnO_4$ to oxidise the camphene to form camphenolide



and camphor after which they are treated with an alcoholic solution of an alkyl nitrate to improve the distribution of the camphenolide and camphor within the goods and finally dried. C. O. C.

Improving the Heat-resistance of Acetylated Cellose. S. *BP* 708,282

The resistance to heat of acetylated cellose containing 2–5 acetyl groups per 3 glucose residues is improved by impregnation with an aqueous solution of dicyandiamide and drying in the normal manner. C. O. C.

Permanently Crimped Yarns of Synthetic Fibres. Heberlein & Co. *BP* 707,859

The yarn is given twist according to the formula $T = \frac{275{,}000}{D + 60} + 800$ (T = turns per metre; D = denier of the yarn) and steam set. It is then untwisted at least to normal twist and, if required, beyond the zero-twist point. C. O. C.

Setting Polyamide or Polyester Fabrics. British Nylon Spinners. *BP* 708,814

The fabric is treated with an aqueous emulsion and/or solution of an organic swelling agent of b.p. $>$ 160°C., if desired together with an electrolyte, and then heated briefly in a dry hot zone at 160°C. up to 10°C. below the m.p. of the fibres. The fabric so set resists dry heat below the temperature of setting used and has improved resistance to creasing even during hot wet treatments. In addition the expansion of the fabric on cooling after leaving the hot zone is less than that which accompanies previously known dry setting processing, the dyeing properties are not impaired and in some cases even deeper dyeings are obtained, and the treated fabrics have very good drape and handle. C. O. C.

Setting and Stiffening Polyamide and Polyester Fabrics. British Nylon Spinners. *BP* 708,815

Fabric consisting wholly or mainly of a synthetic linear polymer is set and/or stiffened by treating it with an aqueous solution of a salt of an amphoteric metal (having a colourless oxide) with a volatile inorganic acid, e.g. $ZnCl_2$, and is then dry set at 10–170°C. below the m.p. of the fibres. C. O. C.

Nylon Plisse. Sayles Finishing Plants. *BP* 708,753

Three dimensional undulating patterns are produced on fabrics wholly or mainly composed of nylon by treating them in localised areas with an aq. soln. of a halogen-substituted aliphatic monocarboxylic acid containing $<$30% acid by wt. The acid is left on the fabric until the treated areas have shrunk to the desired extent. If dyes having affinity for nylon are incorporated in the acid solution brilliant, extremely well penetrated patterns are produced. The treated portions of the fabric have increased affinity for, and may be deeply dyed with direct, acid, acetate, basic or vat dyes. C. O. C.

Coating Polyvinyl Chloride in Patterns on Textiles. Dunlop Rubber Co. *BP* 708,055

A heat-gellable dispersion of polyvinylchloride containing 40–65% of a plasticiser is applied to a fibrous backing through a perforated screen after which the screen is removed and the polyvinyl chloride heated to cause it to adhere firmly to the fabric. If the polyvinyl-chloride is applied in evenly distributed circular spots the fabric remains flexible but has a coefficient of friction $<$ that of leather. C. O. C.

Preventing Runners in Knit Fabrics and Improving the Wearing Properties of Textiles. M. P. Monroe. *USP* 2,643,195

The materials are treated with a solution containing water (7 gal.), granulated potassium alum (116), white corn dextrin (1 oz.), granulated cane sugar (0·5 oz.), magnesium carbonate (0·5 oz.) and sodium chloride (0·25 oz.). C. O. C.

Coating Textiles, Paper, Plastics, etc. with Aluminium by Vacuum Evaporation. National Research Corpn. *USP* 2,643,201

Apparatus in which the base to which the aluminium is to be applied does not become unduly heated and in which the process can be carried on continuously for long periods. C. O. C.

Treating One Substance with Another, One of them at least being a Liquid (I p. 303).
Reducing Shrinkage in Tubular Knitted Fabrics (I p. 303).
Aligning Webs for Lamination (I p. 303).
Emulsion Polymers for the Textile Industry (III p. 304).
Latent Catalysts for Hardening Thermosetting Aminoplasts (III p. 304).
Practical Partial Acetylation of Cotton (VI p. 314).
Disulphide Bond Reactivity in Wool—Formation of Thiol Radicals on Exposure to Light (VI p. 315).
Attack by Moths on Synthetic Fibres in Loose Mixtures and Mixture Textiles (VI p. 316).
Dyeing, Printing, and Chemical Finishing—Recent Developments (VIII p. 319).
Dyeing and Finishing Piece Goods made from Dynel–Rayon and Dynel–Rayon–Acetate Blends (VIII p. 320).
Problems of the Garment Dyer and Finisher, including the Dyeing of Buttons (VIII p. 320).
Bonding Resin-impregnated Cellulosic Webs (XI p. 325).
Fastness to Washing of Crease-resistant Finishes (XIV p. 331).

XI—PAPER AND OTHER CELLULOSIC PRODUCTS

Synthesis of Oligosaccharides by *Chaetomium globosum*. H. W. Buston and A. Jabbar. *Chem. and Ind.*, 48, (9 Jan. 1954).

During a study of the carbohydrate metabolism of *Chaetomium globosum* Kunze, acetone-dried powders and cell-free mycelial extracts of the organisms have been prepared which, when incubated with cellobiose (5% wt./vol.) at 30°C. for 3–4 days produced mainly glucose with a small amount of a trisaccharide which appears to be cellotriose. This appears to be an example of trans-

glycosylation involving a β-link. Incubation with cellobiose (10% wt./vol.) at 30°c. for four days resulted in the almost complete disappearance of cellobiose, the entire disappearance of glucose, and increased production of the trisaccharide, together with higher oligosaccharides so far unidentified. J. W. D.

Acetylation Reactivity of Cellulose containing Salts. C. J. Malm, K. T. Barkey, D. C. May, and P. I. Abell. *Ind. Eng. Chem.*, **46**, 557–561 (March 1954).

Salts such as $NaHCO_3$, Na_2SO_4, $Ca(HCO_3)_2$, $CaSO_4$, $Mg(HCO_3)_2$, and $MgSO_4$ increase the min. time required for activation of an acetylation-grade pulp by acetic acid prior to acetylation. This has been studied using a pulp dried under mild and const. conditions, and with the above salts retained in concn. ranging from 1·2 to 8·8 millimoles per 100 g. pulp; acetylation times and cuprammonium viscosities of the products were recorded at each of four activation times (0, 5, 60, and 120 min.). The effect of the retained salt is related directly to its solubility in acetic acid, and the bicarbonates are soon dissolved and dispersed (their adverse effect diminishing in direct relation), whilst the comparatively insol. sulphates persist and prevent the uniform activation of the pulp for acetylation. The results are tabulated, and shown also in 24 graphs. J. W. D.

Oxidation of Cellobiose by Periodate. F. S. H. Head and G. Hughes. *J.C.S.*, 603–606 (Feb. 1954).

Cellobiose has been oxidised by sodium metaperiodate in the dark at 20°c., and the results indicate that the sugar is oxidised rapidly to an intermediate product with the structure of a formic ester; the subsequent oxidation is slow, because its rate is determined by the rate of the hydrolysis of this ester. The total reaction is represented by the equation— $C_{12}H_{22}O_{11} + 11\ IO_4^- = 9\ H{\cdot}COOH + 2H{\cdot}CHO + CO_2 + 11\ IO_3^-$. These results are shown to be of significance in connection with the side-reactions which occur when periodate acts on cellulose. H. H. H.

Alkaline Degradation of Hemicelluloses—II. V. Prey, E. Waldmann, and W. Krzandalsky. *Sitzungsber. Öster. Akad. Wiss. IIb*, **162**, (8), 888–900 (1953).

Reaction temp. and pH value strongly influence the alkaline degradation of the hemicelluloses of beech-wood, whereas reaction period and amount of alkali (NaOH) per gram of hemicellulose have only slight effects. H. H. H.

Chemistry of Alkali Lignin. V. Prey, E. Waldmann, and F. Stiglbrunner. *Sitzungsber. Öster. Akad. Wiss. IIb*, **162**, (7), 824–827 (1953).

The influence of the hemicellulose content of beech-wood or half stuff upon the amount of alkali lignin obtained during alkali treatment of wood is now reported. The alkali lignins are characterised by mol. wt. and $O{\cdot}CH_3$ and free OH contents. H. H. H.

Alcoholysis of Cellulose. V. I. Sharkov, I. I. Korol'kov, and A. V. Krupnova. *J. Appl. Chem. U.S.S.R.*, **27**, 319–326 (March 1954).

Dry cotton cellulose is treated in an autoclave at 160°c. with solutions of sulphuric acid in methanol, in ethanol, in water, and in water–alcohol mixtures, and the "cellulose content" and degree of polymerisation D.P. (viscometrically after esterification with nitric acid) of the insoluble residue are determined. The residues from the alcoholysis experiments are found to contain up to 15% of humic substances and up to 8% of alkyl groups. The rate of dissolution of cellulose follows a first-order law, and it remains substantially const. in water–alcohol mixtures containing up to 50% of alcohol. With further rise in alcohol content the rate of dissolution increases, and in 100% methanol it is 4·8 times as great as in water (for ethanol the ratio is 3·6). The D.P. of the residue falls from ~1000 to ~115 within a few minutes and then remains const., and this behaviour is independent of the solvent used. It is found also that the D.P.–distribution curves are substantially identical for all of the residues. The effect of the presence of alcohols on the rate of attack is the reverse of that observed in the hydrolysis of sucrose in aqueous–alcoholic solutions, and it is considered that in the heterogeneous reaction there is a multimolecular layer of water at the liquid–solid interface, which acts as a barrier in reactions; only at high concentrations of alcohol is the layer appreciably modified and ultimately transformed into a less effective barrier of alcohol molecules. The water barrier has been shown by Dumanskiĭ to be incapable of dissolving glucose (see *Chem. Abs.*, **28**, 1245 (1934); **31**, 2068 (1937)), and it is now shown by the same method that it cannot dissolve the catalyst (H_2SO_4). In further support of the barrier hypothesis, it is found that, when treatments are carried out in mixtures of 96% or absolute ethanol with benzene, toluene, xylene, or CCl_4, the rate of attack rapidly increases as the proportion of non-polar solvent increases. Further experiments are carried out with acetone and acetone–ethanol mixtures as solvent — reaction is extremely rapid in an acetone soln. of H_2SO_4, and the acetone-insol. residue (which readily gives iodoform when treated with iodine) is completely sol. in cold water.

Experiments are carried out also on mercerised cellulose, which, owing to its increased internal surface, is attacked more rapidly than the original cellulose. Mercerisation is carried out by impregnating the cellulose in 40% or 50% NaOH, pressing to a 200% increase in wt., drying *in vacuo* at 50°c. and then washing with water and again drying. If the dried NaOH-containing material is placed directly in a large vol. of a 10% soln. of H_2SO_4 in 96% ethanol and then transferred to a similar soln. for the alcoholysis treatment, it is found that the rate of reaction is much greater than for the washed mercerised cellulose. This effect is attributed to a further increase in the internal surface of the cellulose due to formation of Na_2SO_4 occlusions among the cellulose macromolecules. A. E. S.

Properties of Chloro Derivatives of Lignin. N. N. Shorygina and L. I. Kolotova. *J. Gen. Chem. U.S.S.R.*, **23**, 2037–2043 (Dec. 1953).

Determinations are made of the contents of Cl, OH, $O{\cdot}CH_3$, COOH, and carbonyl in lignin after treatment under various conditions with chlorine and also after further treatments with water and alkaline solutions. Mild conditions of chlorination (without catalyst at 10°c. in the dark) favour the introduction of non-mobile chlorine atoms. The carbonyl content is little affected by chlorination in CCl_4, but is greatly increased by chlorination in water. The carboxyl content of the chlorinated product is approx. proportional to the chlorine content, and it is increased further in subsequent alkaline (but not neutral) hydrolysis; there is also a great increase in OH content during alkaline or neutral hydrolysis. Some suggestions are made regarding the mechanism of the hydrolysis of chlorolignins. A. E. S.

Tosylation of Hydroxyethyl Celluloses and of Mixed Cellulose Ethers containing Hydroxyethyl Groups. S. N. Ushakov, O. M. Klimova, and É. I. Faingor. *J. Appl. Chem. U.S.S.R.*, **27**, 71–76 (Jan. 1954).

Cotton cellulose (I) is converted into hydroxyethyl cellulose (II) (degree of substitution 0·26, 0·45, and 1·13 hydroxyethyl groups per glucose residue), from which allyl ethers (III) are prepared. I, II, and III are tosylated, and the products are analysed for S and are submitted to the tosyl–iodide exchange reaction in order to determine the primary–hydroxyl contents of I, II, and III. Over the range of substitution examined, introduction of hydroxyethyl groups leads to practically no change in the total number of primary hydroxyls, thus indicating that the etherification proceeds almost entirely at the primary hydroxyls of the cellulose; in the second etherification, reaction again proceeds preferentially at the primary hydroxyls of the two types now present. In order to obtain a mixed ether containing free hydroxyethyl groups, ethyl cellulose is treated with ethylene oxide. The increase in primary-hydroxyl content is much less than that to be expected from the amount of ethylene oxide entering the reaction, and this is attributed to the occurrence of polycondensation with formation of polyether side-chains under the conditions required for reaction at the secondary hydroxyls. A. E. S.

PATENTS

Impregnated Paper. ICI. *BP* 708,622

Impregnated fibrous sheet material, whereof the fibrous sheet has a porous structure to be capable of absorbing throughout the mass of the sheet material

impregnants of particle size up to 2μ., is produced on conventional paper-making machinery and comprises a mixture of fibres of which at least 25% by weight of the dry fibres are substantially non-fibrillated long staple organic fibres, e.g. sisal, hemp, wool or jute, and/or synthetic organic filaments, e.g. rayon or nylon, ranging from ¼ in. to 1½ in. in length and of a difficultly or non-fibrillatable nature and of which the remaining fibres consist of lightly fibrillated organic, e.g. cellulosic fibres, e.g. kraft or lightly beaten, half-rotted cotton, the said fibrous sheet material being impregnated with a natural or synthetic high polymer or polymeric condensation product. The mixture may be 80% rayon, 20% kraft; 50% wool, 50% half-rotted cotton; or 50% rayon, 50% sisal; and the impregnant may be natural rubber, polyvinyl chloride or acetate, or polythene. A deflocculating agent may be added during the dispersion of the fibres later. S. V. S.

Coating of Paper, Cardboard or like Material. N. V. Cellulose Producten. *BP* 707,717

Paper, cardboard or like material is coated by precipitating thereon one or more alkali-soluble cellulose-ethers, e.g. hydroxy ethyl cellulose, with the aid of acid reacting substances, in which the coating layer is washed with water and any remaining acid eliminated with a weakly alkaline substance, e.g. alkali bicarbonate, in which the cellulose ether does not dissolve. Further washing with water may follow until the coated sheet is practically neutral. S. V. S.

Paper of Improved Bleed Resistance to Printing Inks. National Cash Register Co. *USP* 2,641,557

Attapulgite or other clay made up mostly of needle-shaped crystals used as the filter or coating agent yields a paper of improved resistance to ink bleeding. C. O. C.

Coated Paper. Hercules Powder Co. *USP* 2,640,790
USP 2,640,780

A dispersion of a pigment in an aqueous solution of a cellulose ethyl ether β-sulphonate or of a water soluble derivative of a carboxyalkyl hydroxyalkylcellulose is used to impart excellent receptivity to printing. C. O. C.

Bonding Resin-impregnated Cellulosic Webs. Paper Patents Co. *USP* 2,640,799

The sheets are impregnated with a thermosetting resin of the unsaturated polyester type and then pressed together with simultaneous heat curing. C. O. C.

Increasing Surface Friction of Fibres, Fabrics or Films (X p. 322).
Coating Textiles, Paper, Plastics, etc. with Aluminium by Vacuum Evaporation (X p. 323).
Stabilising Carbohydrate Ethers (XIII p. 326).

XII— LEATHER; FURS; OTHER PROTEIN MATERIALS

Divergence in Helical Polypeptide Chains and in Phyllotaxis. A. Frey-Wyssling. *Nature*, **173**, 596 (27 March 1954).

Crystal Structure of Glycyl-L-alanine Hydrobromide. T. C. Tranter. *Nature*, **173**, 221–222 (30 Jan. 1954).

A preliminary analysis is presented of the structure of glycyl-L-alanine hydrobromide, using X-ray methods. J. W. B.

Reaction of Fluorodinitrobenzene with the α- and ε-Amino Groups of Collagen. J. H. Bowes and J. A. Moss. *Biochem. J.*, **55**, 735–741 (Dec. 1953).

The reaction of fluorodinitrobenzene with the amino groups of collagen, modified collagen, and procollagen has been studied. Only 55–60% of the lysine could be recovered as the ε-dinitrophenyl (D.N.P.) derivative from collagen, modified collagen, or gelatin which had been treated with fluorodinitrobenzene. Only small amounts of free amino nitrogen and unchanged lysine were found in the D.N.P. proteins, leaving approx. 30% of the lysine unaccounted for. The possibility of decomposition of ε-D.N.P.-lysine during hydrolysis, and of the presence of acid-resistant ε-D.N.P.-lysine peptides is considered. No terminal residues were detected in collagen, and possible reasons for this are discussed. Small amounts of various end-groups were found in procollagen, the modified collagens, and gelatin. Aspartic acid was most frequently found as a terminal residue, followed by glutamic acid, glycine, alanine, and threonine. With the possible exception of aspartic acid, these are considered to arise from hydrolysis of peptide bonds during the treatments. Either aspartic acid is a terminal residue which is inaccessible to fluorodinitrobenzene or the peptide involving its α-amino group is especially labile. P. G. M.

A Half Century of Progress in Collagen Chemistry. J. H. Highberger. *J. Amer. Leather Chem. Assocn.*, **48**, 704–719 (Dec. 1953).

X-Ray Diffraction Investigation of Swelling of Collagen. M. A. Rougvie and R. S. Bear. *J. Amer. Leather Chem. Assocn.*, **48**, 735–751 (Dec. 1953).

Fibrils of kangaroo tail tendon collagen are swollen in water and studies made of the associated changes in small-angle and equatorial wide-angle X-ray diffraction accompanying graded amounts of water sorption. The total sorption range can be conveniently divided into four intervals— Under rather dry conditions the fibrillar bands are primarily involved, the axes of the kinked, roughly parallel helical chains straightening as moisture is introduced. In the next interval occurs the hydration of polar groups at interbands, whose relatively well ordered, parallel chains begin to separate laterally in orderly fashion. By the third interval the chain-straightening process has proceeded to an extent where small-angle evidence of chain linking continuously disappears. At the same time segments of adjacent chain at bands and interband margins become free of each other. Finally, at equilibrium with pure water the inter-chain separations at interbands once more increase to a spacing of 14–6 A., better oriented equatorially than the corresponding 10–6 A. spacing of the driest material. J. W. B.

Reaction between Chemically Modified Collagen and some Chrome Tanning Complexes. R. L. Sykes. *J. Soc. Leather Trades Chem.*, **38**, 51–58 (Feb. 1954).

Collagens are treated in nine ways, all designed to lower the side-chain carboxyl and/or side-chain amino groups, and are tanned with two chrome liquors, one predominantly anionic and the other predominantly cationic in character. Results indicate no fundamental difference between anionic and cationic tannage, the same reactive groups of collagen being involved. It is also shown that for each particular chromium complex there is a linear relationship between the amount of chromium fixed and the number of ionised carboxyl groups. J. W. B.

Reduction of Amino Acids to Amino-alcohols by Lithium Aluminium Hydride.— II. O. Vogl and M. Pöhm. *Sitzungsber. Österr. Akad. Wiss. IIb*, **162**, (9–10), 1097–1102 (1953).

α-Aminobutyric and α-amino*iso*butyric acids, norleucine, *iso*leucine, α-aminocaproic acid, methionine, α-amino-phenylacetic acid, and tryptophan are readily reduced to the corresponding amino-alcohols by $LiAlH_4$ in tetra-hydrofuran. Tyrosine, lysine, histidine, and glutamic acid, however, behave otherwise. H. H. H.

PATENT

Copolymeric Siloxanes for Rendering Leather Water-repellent. Dow Corning. *BP* 706,719

A compound of formula R_nSiX_{4-n} and/or one of formula $R_nSiO_{\frac{4-n}{2}}$ (R = Alk, monocyclic Ar or halogenated monocyclic Ar; X = Cl or alkoxy; n = 1–3; average degree of substitution being 2·1–3·0 R per Si atom; at least 50% of the R = Alk) is reacted with a silica hydrosol at ≤ 30°C. and pH < 5. There is ≤ 1 mol. organosilicon compound per mol. of SiO_2 in the silica hydrosol. The products applied from solution in a volatile solvent render leather water-repellent without clogging its pores or affecting its ease of polishing, colour or texture. C. O. C.

Automatic Continuous Treatment of Hides by a Series of Processes (I p. 303).
Structures of Basic Chromium Sulphate Complexes (III p. 304).
Precursors of the Red Colour in Leather (IV p. 311).

XIII— RUBBER; RESINS; PLASTICS

Rheology of Solutions of High Polymers. V— Rheology of Very Dilute Solutions. K. Edelmann. *Faserforsch. und Textiltech.*, **5**, 139–145 (April 1954).

It was at one time assumed that the ratio (η_{sp}/c) of the specific viscosity of a high-polymer soln. to its concn. falls asymptotically as $c \to 0$, the extrapolated limiting value being the intrinsic viscosity $[\eta]$ used as a basis for mol.wt. determinations. Early work by Staudinger and by Kern indicated that in the case of polymer–electrolytes η_{sp}/c reaches a min. at an extremely low concn. c_{min}, and then rises again at still lower concn.; an explanation based on electrical effects was advanced. Recently, however, Flory *et al.* (*J. Amer. Chem. Soc.*, **73**, 1904 (1951); *J. Polymer Sci.*, **9**, 381 (1952); **10**, 121 (1953)) have observed a similar phenomenon in non-ionic polymers, and it is now shown that similar behaviour is to be observed at very low concn. ($c_{min} = 10^{-2}$–10^{-3} g./100 c.c.) in solutions of cellulose nitrates in butyl acetate, rubber in toluene, and polyacrylonitriles in dimethylformamide. The higher the mol.wt. of the polymer, the lower is c_{min} and the sharper is the turn in the curve. It must be assumed that at concentrations $> c_{min}$ the effect of immobilisation of solvent becomes more and more important, whereas at concentrations $< c_{min}$ folding of the linear macromolecules is reduced and the groups capable of free rotation become more mobile, with a resulting increase in η_{sp}/c; it is the minimum value of this quantity that should be used in mol.wt. determinations. A. E. S.

Selective Adsorption of Dyes by Synthetic Resins. A. B. Davankov. *J. Appl. Chem. U.S.S.R.*, **26**, 1290–1298 (Dec. 1953).

Further experiments (cf. J.S.D.C., **67**, 292 (July 1951)) on the colouring of finely dispersed synthetic resins with aqueous solutions of dyes show that polyvinyl chloride (as latex or powder) takes up 1–3% of basic dye (direct and acid dyes have no affinity) in a form that cannot be extracted with boiling methanol. The colour of the polymer has a high fastness, not normally associated with the dyes used, to light and to strong solutions of acids and alkalis. A. E. S.

Use of Organic Colouring Matters in Plastics. C. R. M. Oehlcke. J.S.D.C., **70**, 137–145 (April 1954).

PATENTS

Stabilising Carbohydrate Ethers. Brycel. *BP* 707,292

Ethers of carbohydrates which decompose to give aldehydes on heating, e.g. benzyl or ethyl cellulose, are stabilised by incorporating a substance which can react with the aldehyde, e.g. urea, and a catalyst for this reaction, e.g. diphenylguanidine.

BP 707,370

The reactive substance may be an amine, ammonia, hydrazine, hydroxylamine or an acid anhydride. W. G. C.

Plasticised Polyvinyl Chloride. ICI. *BP* 707,235

The esters of a dibasic acid, e.g. phthalic acid, and a mixture of a straight chain and a 2-methyl substituted primary alcohol are useful plasticisers for polyvinyl chloride. W. G. C.

Coloured Polymers of Alkyl Methacrylate. ICI. *BP* 708,124

Coloured products are obtained by placing in a mould a liquid made up of an alkylmethacrylate which may be partly polymerised, a basic dye and a zinc salt and completing polymerisation of the mixture. C. O. C.

Antistatic Finish on Organic Thermoplastic Materials. ICI. *BP* 707,549

The material, e.g. polythene film, is treated with a wetting agent dissolved in a non-solvent for the plastic and in which is dispersed a synthetic organic polymer which is not harder than the plastic and drying so that the applied polymer remains as discrete particles. C. O. C.

Antistatic Coating for Plastics. Nash-Kelvinator Corpn. *USP* 2,640,817

A mixture of a solid, resinous polychlorobiphenyl (30–50%) and an acrylate polymer or copolymer (70–50%) applied in a volatile solvent forms an almost astatic coating on plastics. The effect can be improved by addition of a substance having a free amino group or by using such a substance as one of the components of one of the resins. C. O. C.

Rendering Polyvinyl Acetate More Resistant to Heat, Organic Solvents, Water and Mechanical Deformation. British Oxygen Co. *BP* 706,577

Polyvinyl acetate may be cross-linked by heating it with a polymerisable compound containing > 1 $CH_2{:}CH_2$ group in the molecule, e.g. triallylphosphate. C. O. C.

Decorative Roll of Plastic Material. A. G. L. G. M. M. de Malleray. *BP* 707,014

Several coloured strands of plastic material are formed longitudinally into a roll which is then reduced to the desired diameter. The product retains the design of the original roll but there is some blending with resultant loss of sharpness of outline where one colour meets another. C. O. C.

Basic Linear Polyureas and Polyurethanes (III p. 304).
Problems of the Garment Dyer and Finisher, including the Dyeing of Buttons (VIII p. 320).
Metal Coatings on Plastics (IX p. 321).
Increasing Surface Friction of Fibres, Fabrics or Films (X p. 322).
Coating Textiles, Paper, Plastics, etc. with Aluminium by Vacuum Evaporation (X p. 323).
Rheology of Solutions of High Polymers. IV— Effect of Polydispersity (XIV p. 330).

XIV— ANALYSIS; TESTING; APPARATUS

Determination of Both Cuprous and Cupric Ions in Solution— Application of Citrate Complex Formation. S. J. Quadir and M. H. Khundkar. *J. Indian Chem. Soc.*, **30**, 846–850 (Dec. 1953).

Rapid Estimation of Hardness of Water with Ethylenediaminetetra-acetic Acid. O. Deschler. *Textil-Praxis*, **9**, 183–185 (Feb. 1954).

Sequestering agents such as Trilon B (IG)— ethylenediaminetetra-acetic acid— react with Ca and Mg salts and can be used for rapid and accurate estimation of water hardness. The total hardness (CaO and MgO) is first estimated by titration at pH 10 with Eriochrome Black T (Riedel de Häen) or Chromogen Black (FBy) of analytical quality as indicator, and then the hardness due to Ca is estimated by a second titration at pH 12 with murexide as indicator, the Mg being then calculated by difference. Murexide changes from red-violet to blue, and the two chrome blacks from a bluish red to blue. A more pronounced colour change is obtained by adding Methyl Red to the chrome black, the change then being from a red to green. B. K.

Microdetermination of the Hardness of Water. H. Flaschka. *Melliand Textilber.*, **34**, 1059–1060 (Nov. 1953).

Full directions for the determination of the Ca and Mg hardness of water (5 c.c. sample per titration) by means of a soln. of disodium ethylenediaminetetra-acetate are given together with the theory of the method. H. E. N.

Comparison of the Oxygen consumed from Permanganate and B.O.D. Tests in the Study of Textile Wastes. A.A.T.C.C. Committee on Stream Pollution. *Amer. Dyestuff Rep.*, **43**, P130–P131 (1 March 1954).

Data from different test methods on a variety of effluents are provided, and conclusions drawn as to their suitability in different circumstances. J. W. B.

Simplified Procedure for Water Determination by the Karl Fischer Method. I— Application to Dry-cleaning Detergents and Solvents. A. R. Martin and A. C. Lloyd. *J. Amer. Oil Chem. Soc.*, **30**, 594–597 (Dec. 1953).

The Karl Fischer procedure, as modified by Seaman *et al.* (*Anal. Chem.*, **21**, 510–512 (1949)), has been further simplified so that it may be used as a routine analytical method, using the visible end-point. Some applications to organic solvents and dry-cleaning detergents are described. P. G. M.

Analysis of Textile Auxiliary Products. J. A. van der Hoeve. *J.S.D.C.*, **70**, 145–154 (April 1954).

Since the previous paper (Idem, *Rec. Trav. chim.*, **67**, 649 (1948)) not only have a great many new products been marketed, but new groups of products have also been developed, such as the cation–anion-active compounds and the polyamides (not proteins). Both these new groups behave in many respects like polyethylene oxide compounds, and consequently are included according to the old system among the aminopolyethylene oxides. It has proved desirable to subject this latter group to further subdivision. In addition to this, even before the previous paper a number of groups were known which at the time, owing to lack of samples, had not yet been included in the analytical system, e.g. phosphonium compounds, alkylnaphthalenephosphonates, alkylamidoalkyl phosphates, alkylamidoalkanecarboxylates. The separation of the cation-active amines can now be continued much further than was possible at that time. These factors have induced me to reconsider the analysis of textile auxiliary products, and to attempt to classify them into a larger number of groups by means of the initial tests. AUTHOR

Standardisation of Methods of Test for Auxiliary Products. G. Schwen. *Melliand Textilber.*, **34**, 969–971 (Oct. 1953).

Protection against calcium soap formation is tested as follows— the dispersing agent is dissolved in increasing amounts in dist. water, a soln. of sodium oleate (better than ordinary soap) is added, and aliquot parts of the mixture are stirred into water of known hardness. With small amounts of dispersing agent pptn. will occur, with larger amounts only opalescence. The result can be expressed by interpolation as a percentage of dispersing agent on the wt. of sodium oleate. Results are reproducible. It is shown that in this test, using a polyethylene condensate, up to 50°c., increasing amounts were required, but above this temp. the amount required falls off rapidly. Harder water needs greater concn. of dispersing agent. Some comments are made on the evaluation of other properties. H. E. N.

Evaluation of Modified Starch Products by means of the Iodine Reaction. N. Iwanow and R. Schutz. *Bull. Inst. Textile France* (**42**), 29–41 (Oct. 1953).

A method enabling the heterogeneity of a starch product to be estimated is described. A carefully prepared sample of the starch is introduced into a micro apparatus, stained with iodine, and microscopically examined under polarised light. Increase in dextrinisation results in a change of tint of the starch grains from blue to violet. Temp. and concn. of the iodine soln. must be standardised, and electrolytes, particularly chlorides, should be absent. When starches are slightly modified, only viscosity tests are sensitive. Examination, by means of the iodine technique, of samples similar in viscometric properties, but varying in reducing power, indicated that the sample with higher reducing value was more heterogeneous in composition. G. L.

Spectrophotometric Determination of Cholesterol and Triterpene Alcohols in Wool Wax. F. E. Luddy, A. Turner, and J. T. Scanlan. *Anal. Chem.*, **25**, 1497–1499 (Oct. 1953).

The coloured derivatives of cholesterol (I) and of the triterpene alcohols (II) produced by the Liebermann–Burchard reaction obey the Beer–Lambert law, and a procedure based upon this reaction has been applied to the quant. determination of I and II in wool wax (*Bull. Soc. Chim. biol.*, **27**, 419–424 (1945)). Account is not taken, however, of the appreciable absorption of the coloured derivatives of I in the region in which those of II have their max., and *vice versa*. It is, moreover, difficult to prevent the reaction from proceeding beyond the point of max. colour development while the necessary measurements are being made, and colour development and readings must be conducted at 12°c. Addition of 1:4-dioxan to the reaction mixture reduces the rate of colour development and permits operation at room temp. A spectrophotometer may be used to determine both I and II in the same soln., the former at 630 mμ. and the latter at the secondary max. at 530 mμ. The overlapping of absorption bands is corrected for. Various modifications make the method applicable to the unsaponified wool-wax esters, to the mixture of free alcohols obtained by the saponification of wool wax, or to pptd. cholesterol digitonide. The procedures are fully detailed, and test analyses discussed. J. W. D.

Estimation of Cholesterol and Triterpenols in Unsaponifiable Fraction of Wool Wax. H. Duewell. *Anal. Chem.*, **25**, 1548–1550 (Oct. 1953).

The procedure of Lederer and Tchen (*Bull. Soc. Chim. biol.*, **27**, 419 (1945)) is modified and adapted to allow the simultaneous determination of cholesterol (I) and triterpene alcohols (II). Standard soln. of I and II are used rather than calibration curves, which apply strictly at only one temp., and require very careful control of the composition of the Liebermann–Burchard reagent. Evaporation to dryness (at room temp. to reduce oxidation of the triterpene alcohols) is avoided by using equal vol. of soln. for samples and standards. The developed colours are measured spectrophotometrically and allowance is made for the overlapping of the absorbance curves due to I and II. Trial analyses of known mixtures gave results which were within $\pm$ 3% of the required values. It is probable that 60 μg. of I and 10 μg. of II represent the smallest quantities which can be satisfactorily determined in aliquots. Aliphatic wool-wax alcohols do not interfere. J. W. D.

Determination of Mineral Oil in Textile Oils. M. Antonacci. *Chimica e Industria*, **35**, 561–562 (1953): *Chem. Abs.*, **48**, 4862 (25 April 1954).

The free fatty acids separated from 10 g. of the oil by hydrolysing with HCl and extracting with ether, are saponified with 50 c.c. 0·5N-alcoholic KOH in a water-bath for 2 hr. and the excess KOH titrated with 0·5N-H_2SO_4 against phenolphthalein. The amount of mineral oil is given by the difference between the total free fatty acids and the amount of fatty acid calculated from the KOH used up during saponification (1 c.c. 0·5*N*-KOH is reckoned as 0·145 g. acid where the fatty acid is unknown). In determinations of known mixtures containing paraffin the amount found was —1·10 to + 0·72% of the actual content. C. O. C.

Acetylation of Paper for Chromatography. J. E. Scott and L. Golberg. *Chem. and Ind.*, 48–49 (9 Jan. 1954).

Acidic catalysts cause some degradation of the cellulose chains. A stronger paper is yielded by the method of Hess and Lijubitsch (Dor/e, *The Methods of Cellulose Chemistry* (London: Chapman & Hall, 1946), p. 279). J. W. D.

Paper-chromatographic Separation of Hydrophobic Compounds with Acetylated Cellulose Paper. F. Micheel and H. Schweppe. *Mikrochim. Acta*, 53–63 (1954): *Chem. Abs.*, **48**, 4354 (25 April 1954).

Filter paper acetylated so as to retain its fibre structure is suitable for the chromatographic separation of hydrophobic materials. It is possible to separate and detect microchemically such compounds as isomeric phenols and isomeric aromatic amines and their derivatives, or hydrophobic acetate-rayon dyes. 16 references. C. O. C.

Paper-chromatographic Separation of Chlorophenols and Chlorophenoxyacetic Acids. A. Siegel and K. Schlögl. *Sitzungsber. Öster. Akad. Wiss. IIb*, **162** (7), 686–691 (1953).

The technically important chlorophenols, the chlorophenoxyacetic acids, and also the coupling products (azo dyes) with diazotised sulphanilic acid are shown to be separable by paper chromatography. H. H. H.

Determination of Phenanthrene in Coal-tar Products. L. Blom and W. J. Vranken. *Anal. Chem.*, **26**, 404–407 (Feb. 1954).

A standard procedure is detailed which is a modification of that due to Khmelevskii and Levin (*Org. Chem. Ind. (U.S.S.R.)*, **7**, 241 (1940)). The initial oxidation of phenanthrene to phenanthrenequinone is not effected by the method of the original authors, but by a modification of that of Williams (*J. Amer. Chem. Soc.*, **43**, 1911 (1921)). The active oxidant used is iodine pentoxide and the quinone is caused to react, in $NaHSO_3$ soln., with *o*-phenylenediamine hydrochloride to form the highly insol. phenanthrophenazine; the finish is gravimetric. Carbazole and acenaphthene interfere, but may be removed

prior to the oxidation by pptn. with HCHO. Acridine also interferes, but the effect is negligible at concn. $<5\%$. There is no interference from naphthalene, anthracene, fluorene, pyridine bases, or phenolic compounds (which are pptd. by the HCHO). Results of trial analyses are quoted.
J. W. D.

Interference of Inorganic Salts in the Chromatography on Paper of Amino Alcohols, Diamines, and Diamino Acids. D. M. Waldron-Edward. *Chem. and Ind.*, 104–106 (23 Jan. 1954).

The presence of 5–50 μg. of SO_4^{2+} drastically reduces the R_f values of many amino compounds when paper chromatograms are developed in neutral or mildly acidic or basic solvents; no interference takes place when strongly acidic or basic solvents are used. Local traces of SO_4^{2+} remaining when an acid protein hydrolysate is neutralised with $Ba(OH)_2$ may cause confusion, since they will retard the movement of the compound concerned (which will, however, remain normal in areas where SO_4^{2+} is absent), giving rise to two spots for the same substance, the one slow-moving, the other normal, falsely suggesting the presence of other substances. If sufficient SO_4^{2+} is present, only the slow-moving spots are obtained. The retarding effect of Cl^- is more restricted in scope and magnitude. The interference has been observed with many amino acids and other amino compounds (these being listed), and experimental data are tabulated and discussed showing the effect of various combinations of ions upon the R_f values of ethylenediamine in solvent systems of varying pH.
J. W. D.

Accuracy of Determination of Hydrogen Peroxide by Cerate Oxidimetry. E. C. Hurdis and H. Romeyn. *Anal. Chem.*, **26**, 320–325 (Feb. 1954).

The accuracy of the cerate method is confirmed, and no significant difference is found between results obtained by the cerate and the permanganate methods. The concn. of the cerate soln. does not affect the accuracy, and the substitution of sulphatocerate for nitratocerate is without effect also. The use of reagent-grade cerate is not essential to the attainment of max. accuracy.
J. W. D.

Dye Analysis. IV—Paper-chromatographic Separation of Water-soluble Coulter Dyes. H. Thaler and G. Sommer. *Z. Lebensm. —Untersuch. v. –Forsch.*, **97**, 345—365 (1953): *Chem. Abs.*, **48**, 2376 (28 Feb. 1954).

The following classes and numbers of dyes were investigated: 1 nitroso, 2 nitro, 37 azo, 4 pyrazolone, 18 di- and triphenylmethane, 6 Xanthene, 1 quinoline, 5 azine, 2 thiazine and 1 quinone. Effect of age of the dye soln. on the S_f value, and the effect of various buffers, pH and salts on R_f values were determined. Tables contain the colour in daylight and in ultra violet radiation of the direct chromatographs, and the same after treatment with 3N-NaOH and fuming HCl respectively. Sharp separation of many dyes was not adequately obtained using the common flowing agents, e.g. alcohol, dil. HCl, butanol, etc. 0·1N-NaOH or 2·5% aq. NH_4OH improves separation depending upon the constitution of the dyes. Most dyes showed a relation of R_f to be pH and also to the type of buffer used. Salts in buffer solutions strongly inhibited the movement rate of the dye. Increasing the pH of such salt solutions above neutral point causes considerable increase in the movement rate. Taking advantage of the salting-out of adsorption and the R_f increase by alkali, especially with NH_4OH, permits sharper separation of those dyes with slight differences in behaviour. Satisfactory chromatographs can be got in 1–2 hr. using 2% Na citrate in 5% NH_4OH as standard flowing agent. A procedure is described for investigating food dyes.
C. O. C.

Polarographic Method of Analysis in Control of Production in the Aniline Dye Industry. Y. I. Vainshtein. *Trudy Konissii Anal. Khim., Akad. Nauk S.S.S.R., Otdel. kim. nauk*, **4** (7), 85–95 (1952): *Chem. Abs.*, **48**, 2375 (25 Feb. 1954).

A brief discussion is followed by details of methods for estimating 2:4-disulpho-benzaldehyde, *p*-aminobenzaldehyde and *p*-nitrotoluene in mixtures of the three, and for estimating *p*-methoxybenzoylacetic acid.
C. O. C.

Surface Area Measurement of Paint Pigments. P. C. Carman. *J. Oil and Col. Chem. Assocn.*, **37**, 165–173 (April 1954).

The recording and the comparison of complete distribution curves are more difficult than of single numerical quantities. Size distributions are, in practice, often used merely to calculate mean particle sizes. The quantity most closely related to particle size is the specific surface of the pigment, the most important mean particle size being the "surface mean diameter", i.e. the diameter of a spherical particle with the same specific surface as the pigment. This may be directly determined by three types of procedure—(i) adsorption methods, (ii) permeability methods, and (iii) optical methods. Although they are of high accuracy, methods of type (i) require equipment which is not suitable for routine control work. A simple method based upon air permeability is described in full, and there is a detailed discussion of optical methods based upon the measurement of the emergence intensity of a parallel beam of light passing through a dispersion of the pigment in a liquid medium. The methods are compared, and theoretical reasons are advanced for regarding optical methods as completely untrustworthy in the size range appropriate to paint pigments.
J. W. D.

Meaning and Assessment of Light Fastness in Relation to Pigments. F. M. Smith and D. M. Stead. *J. Oil and Col. Chem. Assocn.*, **37**, 117–130 (March 1954).

There is a brief preliminary discussion of the *B.S.*1006 and the S.D.C. Geometric Grey Scales, and a comparison of carbon arc lamps and daylight as light sources. The effects of pigments concn., formulation, and dispersion are illustrated by data obtained with a Kelvin & Hughes Fade Outfit; these show that the light fastness of a pigment is without significance unless the formulation of which it is a part is specified. Ideally the atmospheric conditions, humidity, and the time and temp. of exposure to a standard light source should also be specified. The adoption of the Blue Wool Scale (*B.S.*1006) in conjunction with the International Geometric Grey Scale is strongly advocated.
J. W. D.

Abrasion Resistance Tester. J. Mecheels. *Melliand Textilber.*, **34**, 848 (Sept.), 950 (Oct. 1953).

A new sample holder for the Frank–Hauser rotary abrasion tester is described. In this, the sample is pressed against the sandpaper (No. 0) by means of a rubber membrane inflated with air at a pressure of 5 g./sq.cm. Results are much more reproducible, and are expressed as the distance travelled at 25% loss in wt. through abrasion.
H. E. N.

Test for Unripe Cotton Fibres. W. Ulmer. *Melliand Textilber.*, **34**, 1055 (Nov. 1953).

The content of unripe fibres in a sample of cotton can be determined by dyeing it with 1·2% Diphenyl Fast Red 5BLN conc. (Gy) + 2·8% Chlorantine Fast Green BLL (Ciba), when ripe fibres become red, the unripe green. Unripe fibres have greater affinity for dyes because of their greater protein content.
H. E. N.

Flax-retting Control. T. Madlung. *Faserforsch. und Textiltech.*, **5**, 112–115 (March 1954).

Sample cuttings of stalk taken in the course of the retting process are split at one end, the divided ends are bent over, and, by the aid of one of these ends, fibre bundles are drawn from the stalk. The fibre bundles are torn from the stalk in an autographically recording rip tester, and the readings taken, which indicate the force required to separate fibre from the wood, enable the progress and the uniformity of the retting to be judged.
A. E. S.

Solubility in Acid as a means of determining Changes in Wool. H. Zahn and A. Würz. *J. Textile Inst.*, **45**, P 88–P 92 (March 1954).

Samples of wool (0·4 g.) are dried for 8 hr. at 105°C. and treated with different concn. of HCl (40 ml.) for 60 min. at 65°C., with shaking every 10 min. After neutralisation to the isoelectric point and thorough washing, losses in weight are determined. It is shown that 4N-HCl is a suitable concn. to give 10–12% weight loss with normal wool, the coefficient of variation (10 samples) being $>$ that in the alkali solubility method (0·1N-NaOH at 65°C.). A large selection of chemically treated wools are examined under these optimum conditions, for both acid and alkali

solubility, and results are tabulated. It is considered that the procedure is of value in conjunction with the alkali solubility test, as it removes many of the uncertainties which arise from the latter alone. J. W. B.

Circular Paper Chromatography— Studies of Physical Factors that may influence *Rf* Values. A. Saifer and I. Oreskes. *Anal. Chem.*, **25**, 1539–1544 (Oct. 1953).

A systematic study is reported of the physical factors which influence R_f values for a simple amino acid system (glycine, alanine, and valine, each in DL form); pH and paper quality were kept constant. Length of exposed wick, and temp. were found to be of greatest influence amongst the numerous factors studied. Rate of solvent flow is best controlled by varying wick width. The method, which has many advantages, gave R_f values with a standard deviation of $\pm$ 0·02 unit or less. No significant changes in R_f values were obtained for such factors as the presence of the inverse phase, equilibration of the paper with solvent vapour prior to running the chromatogram, variation of wick width (2–8 mm.), time of running (5–48 hr.), concn. of amino acid (5–25 μg.), and the freshness of the phenol used. The application of the method to complex amino acid systems is under investigation. J. W. D.

Detection of Tin in Weighted Silks. F. Feigl, V. Gentil, and D. Goldstein. *Mikrochim. Acta*, 93–95 (1954): *Chem. Abs.*, **48**, 4362 (25 April 1954).

Heat a sample of the ash in a tiny test tube with 3 drops of conc. H_2SO_4 for about a min. Complete dissolution is unnecessary. Cool, add an equal volume of water and some KI from a knife blade, and shake with benzene containing iodine. Place a drop of the benzene solution on filter paper, allow to drop and treat the spot with NH_3. Then add in succession one drop of 5% Na_2SO_3 and a drop of morin solution; treat with acetic acid. If tin is present a greenish blue fluorescent spot can be seen in ultraviolet radiation. C. O. C.

Determination of the Sequence of Amino Acids in Peptides and Proteins.

II— Separation of the Methyl Esters of *N*-2:4-Dinitrophenyl Derivatives of Amino Acids by Adsorption Chromatography and the Free Amino Groups of Insulin. C. M. Fletcher, A. G. Lowther, and W. S. Reith. *Biochem. J.*, **56**, 106–111 (Jan. 1954).

Methyl esters of *N*-2:4-dinitrophenyl (D.N.P.) derivatives of a number of amino acids have been prepared. The separation of mixtures of these derivatives by adsorption chromatography on alumina columns was investigated. The stability to acid hydrolysis of some D.N.P.-amino acids was investigated by two different methods. The new method of separation of D.N.P.-amino acids (by adsorption chromatography of their methyl esters) was applied to the re-investigation of the free amino groups of insulin. Assuming a mol.wt. of 12,000, it is shown that two free amino groups are on phenylalanine residues, two or three on glycine residues, and two represent the ε-amino groups of lysine. In addition, a new, unidentified substance bearing a free amino group was found. It is suggested that, owing to the difficulty of an exact assessment of the magnitude of correction factors for the decomposition of D.N.P.-amino acids during protein hydrolysis, it must be considered as quite uncertain how far the calculated values for the amount of *N*-terminal amino residues really correspond to the amount actually contained in insulin.

III— Synthesis of 3-(4-Dimethylamino-3:5-dinitrophenyl)hydantoin Derivatives of Various Amino Acids, and their Use for the Determination of *N*-Terminal Amino Acids. G. G. Evans and W. S. Reith. *Ibid.*, **56**, 111–116 (Jan. 1954).

Red 4-dimethylamino-3:5-dinitrophenyl *iso*cyanate (D.D.P.I.) was synthesised. It was found that this *iso*cyanate reacts quantitatively with the free amino groups of amino acids or peptides dissolved in water–dioxan at the apparent pH 9·6 at 0°c., giving deep orange carbamoyl derivatives (D.D.P.-ureidoamino acids or D.D.P.-ureidopeptides). D.D.P.-ureidoamino acids when refluxed for 2 hr. in 2*N*-hydrochloric acid, or when treated for several days at 40°c., with nitromethane saturated with hydrochloric acid, give by ring closure and elimination of water the corresponding hydantoins (amino acid-D.D.P.-hydantoins) in quantitative yields. When D.D.P.-ureidopeptides are treated in the same way, the *N*-terminal amino acid carrying the D.D.P. radical splits up, forming by ring closure the corresponding hydantoin, which can be recovered in quantitative yields. The amino acid-D.D.P.-hydantoins are deep orange substances which crystallise well and have sharp melting points. They can be adsorbed on alumina columns, and their mixtures can be conveniently resolved by adsorption chromatography. The scope for the use of these derivatives for the determination of *N*-terminal amino acids in peptides is discussed.

IV— Synthesis of 3-(4-Dimethylamino-3:5-dinitrophenyl)-2-thiohydantoin Derivatives of Various Amino Acids, and their Use for Amino Acid Sequence Determinations. W. S. Reith and N. M. Waldron. *Ibid.*, **56**, 116–120 (Jan. 1954).

Deep orange 4-dimethylamino-3:5-dinitrophenyl *iso*thiocyanate (D.D.P.T.) was synthesised. It was found that this *iso*thiocyanate reacts with amino acids or peptides dissolved in water–acetone (1 : 3 by vol.) at the apparent pH 8–9, giving orange thiocarbamoyl derivatives (D.D.P.-thioureidoamino acids or D.D.P.-thioureidopeptides) in practically quantitative yields. By treatment of the D.D.P.-thioureidoamino acids with aqueous or glacial acetic acid the corresponding substituted 2-thiohydantoins (D.D.P.-thiohydantoins) are formed in good yields (80–90%). By treatment of D.D.P.-thioureidopeptides with aqueous acetic acid, the *N*-terminal amino acid residue splits off as an amino acid-D.D.P.-thiohydantoin, which can be isolated in a pure state by adsorption chromatography; the residual peptide can be treated with D.D.P.T., and the process repeated to determine the sequence of amino acids in the peptide. As an example the sequence determination on alanylglycylglycine is described. The amino acid-D.D.P.-thiohydantoins can be hydrolysed to the parent amino acids, and these can be identified by the use of paper chromatography. P. G. M.

Synthetic Fibres— Properties and Microscopical Identification. W. Bobeth. *Faserforsch. und Textiltech.*, **5**, 115–130 (March), 168–170 (April 1954).

The physical properties of various synthetic fibres and their behaviour in various solvents and swelling media are described, and a detailed discussion is given of the microscopical identification of the fibres, particularly on the basis of their behaviour in swelling media (e.g. formation of a quasilumen, often having characteristic folds, striations, etc.; also peculiar formations at cut ends; cf. J.S.D.C., **69**, 315 (Aug. 1953)). There are many photomicrograph illustrations. A. E. S.

Qualitative Analysis of Synthetic Fibres. M. Lundegard and E. D. Roseberry. *Amer. Dyestuff Rep.*, **43**, 93–97 (15 Feb. 1954).

Simple and accurate methods for identifying synthetic fibres are described, using nylon, Terylene, Orlon, Acrilan, Saran, and polyethylene filament, and dynel and X51 staple fibres. The study is divided into three parts, concerning respectively solubility in different reagents, microscopy, and miscellaneous tests, the latter part including the use of an identification dye (Texchrome), a flame test, a chlorine test, and the Liebermann–Storch reaction to distinguish vinyl acetate, vinyl acrylate, vinyl chloride, and vinylidene chloride. A scheme is presented whereby Orlon, Acrilan, X51, dynel, Saran, polyethylene, nylon, and Terylene dissolve in the reverse order when treated successively with boiling 40% NaOH; cold 80% phenol; CCl_4, monochlorobenzene, acetone, and 50% $ZnCl_2$ all for 10 min. on a steam bath; and 70% H_2SO_4 for 40 min. on a steam bath. Only Orlon survives all these treatments. J. W. B.

Viscometric Determination of the Degree of Polymerisation of Perlon in Concentrated Formic Acid. R. Bennewitz. *Faserforsch. und Textiltech.*, **5**, 155–158 (April 1954).

The relationship used by Loepelmann (see J.S.D.C., **68**, 234 (June 1952)) for the accurate determination of the intrinsic viscosity of solutions of Perlon in *m*-cresol applies also to solutions in formic acid, the required value of m being 1·5. In such solutions, polymer degradation is very slow at room temp., and they are very satisfactory for use in determinations of degree of polymerisation; at higher temp., however, rapid degradation occurs. A. E. S.

Carpet Soiling. K. L. Whitney and J. W. Schappel. *Amer. Dyestuff Rep.*, **43**, P143–P147 (1 March 1954).

Theories of carpet-fibre soiling are reviewed, and experiments briefly described in which the effects of oil content of both pile yarns and jute backing, and of finishing agents, are studied. Particular attention is paid to the behaviour of a new viscose rayon fibre which has a smooth surface. It is less sensitive to soiling and in many ways resembles wool in this respect. J. W. B.

Antistatic Agent Evaluation. W. Sprenkmann. *Melliand Textilber.*, **34**, 971–972 (Oct.), 1076 (Nov. 1953).

A crude test for classifying antistatic agents (for Perlon) into 3 groups is described. The yarn is rubbed over a rod (metal or synthetic) in a const. manner. The fibres, in consequence of electrification, will tend to separate, and the greatest separation in mm. is noted and expressed as a percentage of the separation of rubbed, untreated fibres (for Perlon ca. 140 mm.). A value of up to 10% was considered good, up to 50% useful, on Perlon. H. E. N.

Ironing Tests. K. Schicktanz. *Melliand Textilber.*, **34**, 1144 (Dec. 1953).

The precautions listed should be observed if reproducible results are desired. H. E. N.

Characterisation of Occupational Hazards arising from the Presence of Carbon Disulphide. H. Demus. *Faserforsch. und Textiltech.*, **5**, 65–67 (Feb. 1954).

According to circumstances, either the CS_2 content of the atmosphere or that of the urine can be used to characterise the health hazard to a worker due to the presence of CS_2. Simple methods are described for the former determination (and that of the H_2S content); methods for the latter have been described previously (see J.S.D.C., **69**, 219 (June 1953)). A. E. S.

Differential Refractometer for Coloured Solutions. E. Atherton and E. Cowgill. J.S.D.C., **70**, 116–120 (March 1954).

A differential refractometer, suitable for the measurement of refractive indices of dyes, is described, and some of its uses are indicated. The instrument is of the prism type, utilising a small spectrometer to provide monochromatic light. Refractive index values are obtained by means of a rotating mirror driven by a differential screw, which cancels the deviation conferred on the light beam by the dye solution. Several advantages of the instrument for measurements in spectral regions of maximum absorption are discussed. C. J. W. H.

Laboratory Skein-dyeing Machine. W. Armfield. J.S.D.C., **70**, 183–185 (May 1954).

A convenient laboratory dyeing machine is described for the dyeing of skeins. Dyeings can be carried out in liquor : yarn ratios as low as 10 : 1 using 5 g. of yarn. Twenty-four dyeings can be done simultaneously at the same temperature, or a maximum of twelve at each of two temperatures. Thermostatic temperature control is employed, together with continuous agitation of the yarn, which is kept completely submerged. The advantages compared with the usual pot dyeing methods are listed. AUTHOR

Determination of Fastness to Washing. H. Mendrzyk. *Melliand Textilber.*, **34**, 957 (Oct. 1953).

Tests according to DIN 53,953 and 53,956 may be misleading in cases where faults have been caused by migration of dye during drying. Contrary to these tentative standards, significance is therefore attached to bleeding into the test liquor. H. E. N.

Fastness to Perspiration—II. Society of Dyers and Colourists, Perspiration Fastness Subcommittee. J.S.D.C., **70**, 63–68 (Feb. 1954).

Specific Method for the Determination of Carbonyl Groups in Oxycelluloses. E. D. Kaverzneva and A. S. Salova. *J. Anal. Chem. U.S.S.R.*, **8**, 365–369 (Nov.–Dec. 1953).

Carbonyl contents of oxycelluloses determined by the hydroxylamine method of Gladding and Purves (*J. Paper Trade*, **116**, 26 (1943)) are too high, since, under the conditions used for oxime formation (pH 5–6), hydroxamic acid groups are formed from lactone groupings that are present (see J.S.D.C., **68**, 329 (Aug. 1952)). It is shown that, if the determination is carried out at pH 3–4 and alcohol is used instead of water as medium, only carbonyl groups react. Except in the case of cellulose oxidised with NO_2, it is not generally necessary to neutralise COOH groups present with calcium acetate, and the modified method is as follows—The reagent is prepared (fresh for each determination) by dissolving hydroxylamine hydrochloride (5 g.) in a mixture of 96% alcohol (240 ml.), water (5 ml.), and 0·2% alcoholic Bromophenol Blue (6 ml.), and then adding 0·2 N-NaOH dropwise until the soln. is green to transmitted and red to reflected light; the pH of the soln. (3·2–3·4) is checked potentiometrically. A weighed amount (w = 2–3 g.) of the oxycellulose is covered with 60 ml. of reagent (room temp.), and after 2 hr. the supernatant soln. is filtered and 50 ml. of it is titrated with 0·1 N-NaOH back to pH 3·2 (a ml.). The carbonyl content (%) is given by—

$$\frac{a \times 0{\cdot}0028 \times 1{\cdot}2 \times 100}{w}$$

A. E. S.

Determination of Iron in Vegetable Tanning Extracts. G. Forsyth. *J. Soc. Leather Trades Chem.*, **38**, 8–10 (Jan. 1954).

A procedure is recommended for determining iron, in which solid or liquid extract is ashed, dissolved in HCl, evaporated, redissolved in HCl and water, and reprecipitated with ammonia. The precipitate is filtered and dissolved in HCl, and an aliquot is mixed with KCNS and compared in a Nessler tube with a standard tube, which is brought to exact match by adding standard iron solution (from ferrous ammonium sulphate) to KCNS. J. W. B.

Comparison of Methods for Determination of Percentage Basicity of One-bath Chrome Liquors. D. Burton and M. C. Thompson. *J. Soc. Leather Trades Chem.*, **38**, 102–105 (March 1954).

Particle Size Determinations of Leather-finishing Agents. E. Atherton and R. H. Peters. *J. Soc. Leather Trades Chem.*, **38**, 22–30 (Jan. 1954).

A procedure is described whereby particle size in aqueous dispersions of resins is measured optically, using a refractometer and an absorptiometer capable of turbidity measurement. The method is suitable for routine determinations. J. W. B.

Rheology of Solutions of High Polymers. IV—Effect of Polydispersity. K. Edelmann. *Faserforsch. und Textiltech.*, **5**, 59–64 (Feb. 1954).

Available methods of determining the polydispersity of a soln. of a high polymer depend on the application of fractionation techniques (the most accurate), or on a comparison of the results of viscometric and osmotic mol.wt. estimations; they are too complex and time-consuming for general use. It is shown that a measure of polydispersity that is adequate for many purposes can be obtained from the results of 3–5 measurements of shearing stress at various flow gradients. The author has previously analysed the flow curves of such solutions (see J.S.D.C., **69**, 106 (March 1953)), and he now shows that a single curve characterising the flow behaviour of solutions of all concentrations in a given solvent can be obtained by plotting—

$$\frac{\ln\ \eta/\eta_\infty}{\ln\ \eta_0/\eta_\infty}$$

against $\ln\ v$ (η is the viscosity of a given soln. at flow gradient v; η_0 and η_∞ are the viscosities of the same soln. at low and high shearing force respectively—on the lower and upper linear regions respectively of the S-shaped curve previously discussed (*loc. cit.*)). The lower the slope of this curve, the greater the polydispersity, and as a measure of the latter a number inversely proportional to the slope is suggested. A. E. S.

Chromatographic Separation of Sugars with Hydrocellulose. J. D. Geerdes, B. A. Lewis, R. Montgomery, and F. Smith. *Anal. Chem.*, **26**, 264–266 (Feb. 1954).

Dissolution of cellulose powder in 85% H_3PO_4, followed by re-pptn. with water, yields a hydrocellulose whose capacity and resolving power as a chromatographic absorbent for methylated sugars is superior to other cellulose absorbents; no advantage obtains with free sugars. The preparation and construction of the column

are detailed and there is a diagram of the head of the column, a feature of which is a glass-enclosed metal weight which keeps the column in compression, and which is considered to contribute significantly to the effectiveness of the method. Data are tabulated showing the superiority of the hydrocellulose column over one of cellulose itself in the separation of mixtures of methyl glucoses, methyl ethyl ketone–water being used to develop the hydrocellulose column. The synthesis and resolution (difficult by normal classical methods) of α- and β-ethyl-L-rhamnofuranosides is described in full. The method appears also to be valuable in the separation of the cleavage products of methylated polysaccharides. J. W. D.

Determination of Total and Primary Hydroxyl in Cellulose Esters by Ultraviolet Absorption Methods. C. J. Malm, L. J. Tanghe, B. C. Laird, and G. D. Smith. *Anal. Chem.*, **26**, 188–190 (Jan. 1954).

Different amounts of primary and secondary OH groups are formed during the hydrolysis of cellulose acetate, depending on the exact conditions of hydrolysis. Phenyl *iso*cyanate reacts rapidly and completely with the OH groups in hydrolysed cellulose acetate in pyridine soln. at 100°C. to form a carbanilate (*N*-phenylcarbamate) which can be determined by means of its absorbance at 280 mμ. This affords a measure of the total OH groups (primary + secondary). Primary OH groups may be determined by tritylation using trityl chloride in pyridine soln., followed by measurement of the absorbance at 259 mμ. The methods are given in full detail, and spectrophotometric curves are shown. A wide variety of cellulose esters has been examined by these methods, and a typical cellulose acetate has been used to determine their precisions; this work is reported. There is good agreement between the results of carbanilation and saponification, and of tritylation and recovery of the triphenylcarbinol by pptn. from conc. H_2SO_4 soln. by water. J. W. D.

Gloss Measurement of High-gloss Papers. V. G. W. Harrison and S. R. C. Poulter. *Research*, **7**, 128–132 (April 1954).

Gloss is not a simple physical property, but a psychological *Gestalt*, i.e. an appraisal of a physical situation taken as a whole. The various factors of this physical situation are weighted differently not only by different observers under the same conditions but by the same observer under different conditions. Consequently gloss is *not* a property independent of the observer and his surroundings. The difficulty of obtaining satisfactory agreement between observers increases as the gloss of the surfaces increases and as the colours and surface textures of the papers compared diverge. Thus it is impossible to establish any gloss scale satisfactory to all observers under all conditions of illuminating and viewing. At best a convention can be set up which to be reasonably satisfactory to most observers under average commercial conditions of judging papers. Individual disagreements with this convention will be inevitable and frequent. C. O. C.

Rapid Determination of Resin in Dissolving Pulp. I. Jullander and B. Olsson. *Svensk Papperstidning*, **57**, 151–158 (15 March 1954).

The Swedish standard method for resin determination prescribes 6 hours' extraction time for each solvent, and even if only one solvent is used, the total time for analysis amounts to 10–12 hr. in practice. The Soxhlet extraction with ethanol can be replaced by shaking with warm ethanol for 5 min., and wet pulp from the mill can be extracted directly without previous drying. The dye Rhodamine B is absorbed by pulp from aqueous solution in an amount closely proportional to the amount of extract, and a colorimetric method for quantitative determination of the resin content based on this observation has been developed for dissolving pulp; it takes 30–45 min. S. V. S.

Quantitative Determination of Carbonyl Groups in Oxycellulose by means of Sodium Borohydride. B. Lindberg and O. Theander. *Svensk Papperstidning*, **57**, 83–85 (15 Feb. 1954).

Determination of carbonyl groups in periodate oxycellulose by the Na borohydride method gave a value 4% lower than that calculated from the periodate consumption. This difference may be partly ascribed to the presence of carbonyl groups in the original cellulose and to overoxidation. S. V. S.

Hydrogen Peroxide Determination in the Presence of Chromate. J. Rynasiewicz. *Anal. Chem.*, **26**, 355–358 (Feb. 1954).

Both CrO_4^{2-} and H_2O_2 are adsorbed anionically on ion-exchange resins at high pH. A resin which is effective at nearly neutral conditions will adsorb CrO_4^{2-}, allowing the H_2O_2 to pass; it may then be determined by the pertitanic acid method. The method, including the preparation of the adsorbent resin, is given in full, and numerous results of trial analyses are discussed. The rate of H_2O_2 decomp. in slightly alkaline soln. of Na_2CrO_4 is dependent upon temp., pH, and CrO_4^{2-} concn. J. W. D.

Determination of Yellow Colour in Raw Rubber Latex Films and Crêpes. G. A. Kidder. *Anal. Chem.*, **26**, 311–315 (Feb. 1954).

The natural yellow colouring matter in dry rubber or latex film may be determined by the spectrophotometric examination of an acetone extract of the sample. Absorbances are measured at 360, 440, and 550 mμ., and these are combined in an empirical equation from which is derived the "colour index"—

$$\text{Colour index} = a_{440} - 0{\cdot}2a_{360} - 0{\cdot}8a_{550}$$

The determination is not affected by the darkening of the rubber film by oxidation, since this produces an absorption max. at $<$ 340 mμ. There is also a shift towards shorter wavelengths of the normal peaks by 7–10 mμ., and this is considered to support the view that the yellow colouring matter in natural rubber is a carotenoid closely related to, if not identical with, α-carotene. J. W. D.

Fastness to Washing of Crease-resistant Finishes. M. Sorkin and W. Ernst. *Textil-Rund.*, **9**, 23–24 (Jan. 1954).

The pattern, cleaned with water (40:1, 90 ± 2°c., 5 min.) and dried, is treated in 3% HCl (40:1, 60 ± 2°c., 3 min.), washed with water and dried. The resin is removed completely with 3% HCl (90 ± 2°c., 6 min.). In the first treatment cotton loses 0·15% of its wt., in the second 0·6%. Taking this into account, from the corrected percentage loss in wt. in the first treatment (A) and in the second treatment (B), the percentage washfastness (W) is given by $W = 100\,B/(A + B)$. The following values for W were obtained: Zeset S (DuP), 0–10%; urea–formaldehyde, 0–10%; etherified urea–formaldehyde, up to 30%; lightly etherified melamine–formaldehyde, 30–40%; highly etherified melamine–formaldehyde, 60–75%. Greater amounts of resin give greater W. H. E. N.

Single-fibre Stress–Strain Apparatus. H. M. Preusser, R. A. O'Connell, A. S. Yeiser, and H. P. Lundgren. *Text. Research J.*, **24**, 118–122 (Feb. 1954).

A desk-type, autographically recording instrument for precise stress–strain measurements of single fibres is described. Testing is carried out in a horizontal plane to facilitate measurements in solvent media. Applications to fibre creep measurement and to surface friction measurement are described. A. B.

Method of Measuring Ultrafine Glass Fibres. R. K. Ladisch, B. McQue, and S. L. Knesbach. *Anal. Chem.*, **26**, 399–400 (Feb. 1954).

The free space in bundles of very fine glass fibres often approaches 100%. The surface area per unit weight, which is important in classifying such materials, may be determined by the N_2 adsorption method due to Brunauer, Emmett, and Teller (literature references are given), with a precision of 2% or better. As the vol. ratio of dead space to N_2 adsorption becomes appreciably >1, the precision decreases; the values are still precise to better than 5% for fibres below 2 μ. in diameter. Small deviations in the average fibre diameter between different grades of fibres are readily detected, since the surface area of a given wt. of sample increases inversely with decreasing fibre diameter; relatively coarse fibres in a batch are similarly revealed. The method is detailed. J. W. D.

Polarographic and Amperometric Mercurimetric Determination of Disulphide Groups— in Cystine, Oxidised Glutathione, and Proteins. W. Stricks, I. M. Kolthoff, and N. Tanaka. *Anal. Chem.*, **26**, 299–303 (Feb. 1954).

Disulphide groups in amino acids, peptides, denatured proteins, and acid protein hydrolysates may be determined with an accuracy of ~2%, by mercurimetric titration in alk. soln. in presence of Na_2SO_3; the end-point may be detected either amperometrically, or polarographically at a dropping Hg electrode. The complete hydrolytic disintegration normally required prior to disulphide determination by existing methods is replaced by a more rapid procedure involving pepsin digestion and subsequent partial hydrolysis in M–HCl. The method as a whole is more rapid and accurate than those in current use. Details are given of procedures for the determination of disulphide in cystine, oxidised glutathione, and denatured and partly hydrolysed protein. J. W. D.

Amperometric Mercurimetric Titration of Thiol Groups in Biologically Important Substances— at the Rotated Platinum Wire Electrode as Indicator Electrode. I. M. Kolthoff, W. Stricks, and L. Morren. *Anal. Chem.*, **26**, 366–372 (Feb. 1954).

Thiol groups in cysteine and glutathione can be determined by mercurimetric titration in aq. media, using a rotated Pt wire electrode as indicator electrode. Accuracy and precision of ±2% can be attained in borax, phosphate, carbonate, and ammonia buffers in the range 9–13 pH at Cl^- concn. $<0{\cdot}5$M. Sharp end-points are not obtained at concn. $> 6 \times 10^{-4}$M. with glutathione, and $>10^{-4}$M. with albumin, or at pH >9·2 with cysteine. Procedures are detailed for the rapid and accurate determination of traces of thiol in amino acids, peptides, and in native and denatured proteins. With cysteine the method has no advantage over argentimetry, but with proteins it permits the determination of thiol groups in presence of ammonia (unlike the argentimetric method) and is applicable over a wider range of pH. The method is inapplicable in presence of SO_3^{2-}, but mercurimetric titration may be carried out at a dropping Hg electrode indicator electrode. J. W. D.

Rotating Platinum Electrode for Amperometric Titration of Thiol Groups. F. J. Herbert and J. R. Denson. *Anal. Chem.*, **26**, 440 (Feb. 1954).

A rotating Pt electrode is described (with diagram) that can be operated at higher speeds than most of those in current use; this ensures sharper end-points. Satisfactory contact is achieved at these higher speeds, and since the construction is of glass there are no electrical interferences due to imperfect insulation of the motor from the electrode. J. W. D.

PATENTS

Flammability Tester. Society of the Plastics Industry. *USP* 2,636,388

In apparatus of the type where the material to be tested is placed at an angle over a flame, the timing of the test is automatically controlled. The rack in which the sample is placed holds it firmly in position without the use of supporting wires and so prevents transmission of heat by the supports which is a defect of many previous devices of this type. C. O. C.

Detecting Irregularities on the Surface of Cloth, Paper or the Like. Robbins Mills. *USP* 2,636,951–2

The cloth is passed beneath rings through which a shaft runs, the shaft being smaller in diameter than the internal diameter of the rings. The rings just touch the surface of the cloth and so will be raised slightly by any projections on its surface. When raised they operate an electric circuit which either activates a warning device or stops the machine. C. O. C.

Universal pH Indicator. American Cyanamid Co. *USP* 2,643,230

An indicator capable of determining the pH of leather, cloth and paper in the range pH 1–9 is obtained by mixing slightly alkaline solutions of bromothymol blue and *m*-cresol purple at room temperature using twice as much blue as purple and taking care that no undissolved dye is present. To this mixed solution is added a hot saturated aqueous solution of rosolic acid using about 0·03% of the weight of rosolic acid on the weight of the purple. The whole is then diluted with pure water to 0·04% dye content. The colour of the indicator varies from red at pH through orange to yellow to green to reddish blue at pH 9. C. O. C.

Measuring the Moisture Content of Fabrics. Firestone Tyre & Rubber Co. *BP* 706,487

One pole of a D.C. source (ripple free and isolated from earth) is connected through the fabric to earth. The other pole is connected to earth through a known resistance, the potential difference across which is measured to give a measure of the leakage from the D.C. source through the fabric to earth. C. O. C.

Determining Moisture Content of Fibrous or other Materials. J. L. Shaw. *BP* 709,177

The spaced needles of a needle electrode system are inserted into the material, the capacitance of the material between the needles measured and this measurement converted into an indication of the moisture content. C. O. C.

Reactions of 4-Thionchromones with Amino Compounds, and with Methyl Iodide (IV p. 310).
Microstructure of Paint Films (V p. 313).
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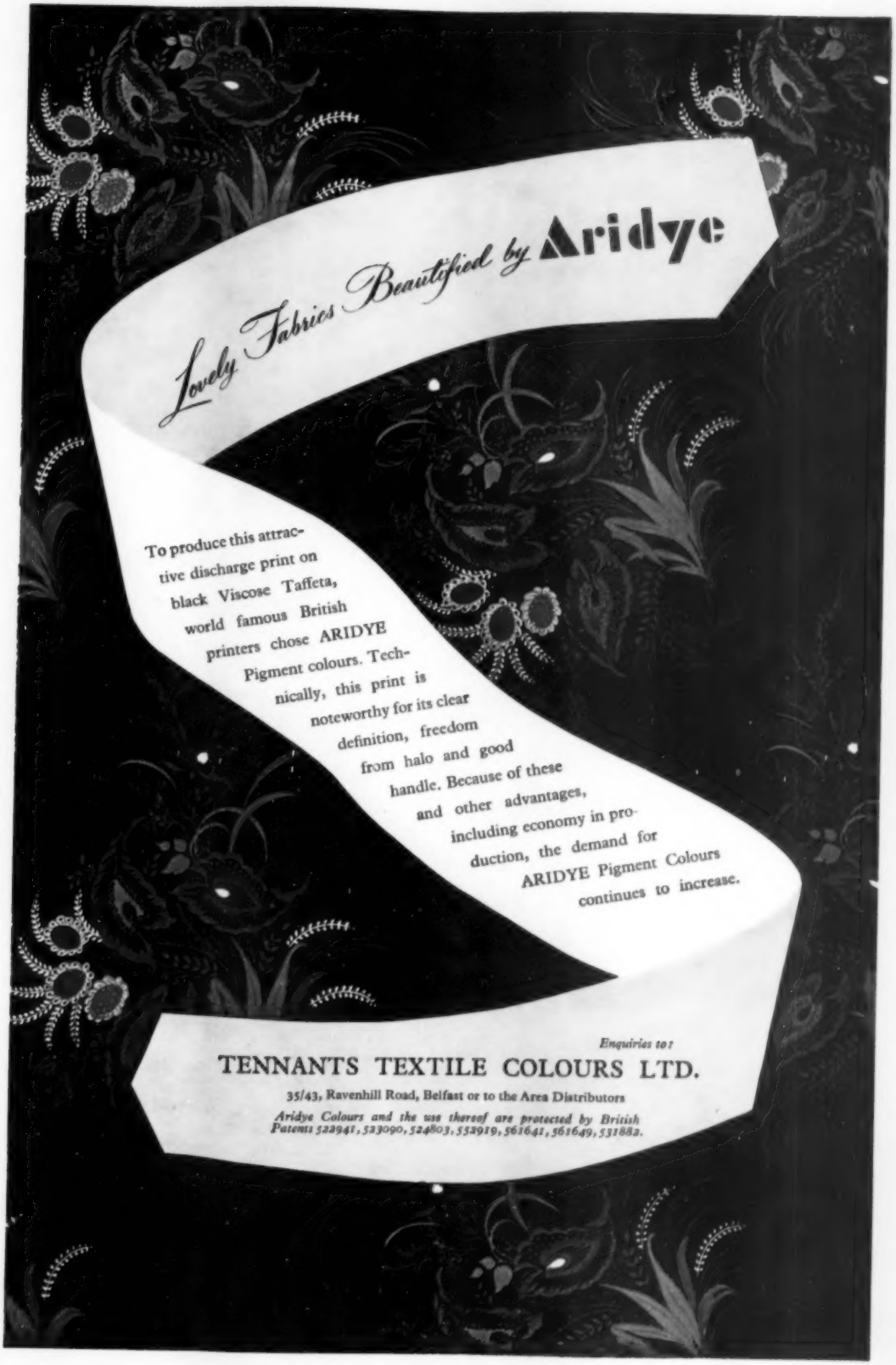
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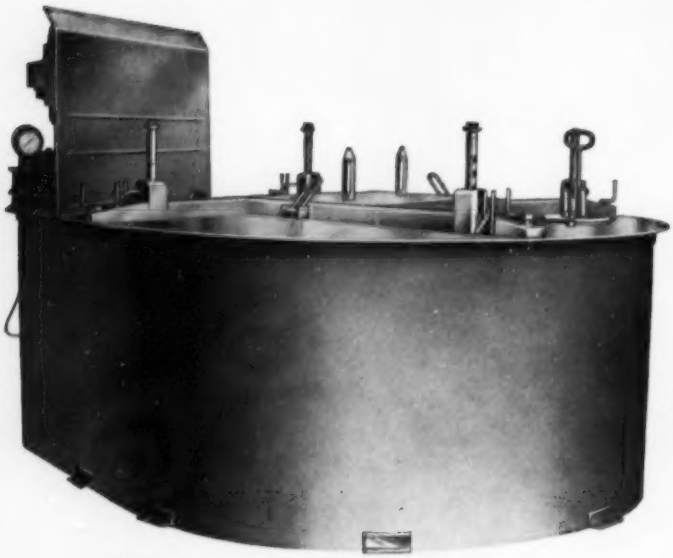

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IMPERIAL CHEMICAL INDUSTRIES

Dr. Alexander Fleck on Proposed Profit-Sharing Scheme

The twenty-seventh annual general meeting of Imperial Chemical Industries Limited was held on June 17th in London.

Dr. Alexander Fleck, the chairman, presided and in the course of his speech, said —

The sales in 1953 were the highest in our history, and with new plants coming into operation we can confidently look forward to further increases in the years to come.

PHARMACEUTICAL INDUSTRY

In the Annual Report reference is made to the attitude of the Ministry of Health towards the cost of pharmaceuticals required by the National Health Service. Any effort by a Government Department to cut down waste and extravagance is commendable. In this instance, however, there is the serious danger that a rather unintelligent application of quite inappropriate costing methods may damage or even destroy the most vital parts of the pharmaceutical industry in this country. The danger is that pharmaceutical research may be rendered so unattractive in this country and so unprofitable that this important field of research may be abandoned by one Company after another, and this country may be forced to rely upon the scientific achievements of other countries. Costing principles permitting a very limited percentage of profit on the manufacturing cost of a commodity may no doubt be appropriate and proper in some cases, but I suggest that in the case of drugs which it is decided to produce and which the National Health Service wants, the rigid and unimaginative application of such principles will lead to results that are unfair to the industry.

PROFIT-SHARING SCHEME

The references in the Report and in the Press to the proposed Profit-Sharing Scheme for employees will not have escaped your notice.

The Scheme provides for an increase in the bonus if dividends are increased. Stockholders and employees will thus both share in any increased prosperity which it may be our good fortune to enjoy in future years. They will also have some share in any adversity which we may suffer.

Bonuses under the Scheme are in no way part of the terms and conditions of employment and are not intended to be a substitute for fair wages and conditions.

So far this year the Company has enjoyed, in common with other industries in this country, a period of expansion, and the prospects are good in general.

The Report and Accounts were adopted.

COURTAULDS' SCIENTIFIC AND EDUCATIONAL TRUST FUND

Entrance Scholarships in Textile Technology

A limited number of Entrance Scholarships, for which funds have been provided by the Courtaulds' Scientific and Educational Trust, tenable at the University of Leeds or at the Manchester College of Technology (University of Manchester), are open to students wishing to pursue studies in any branch of textile technology.

The value of the Scholarships will be £50 per annum for three years.

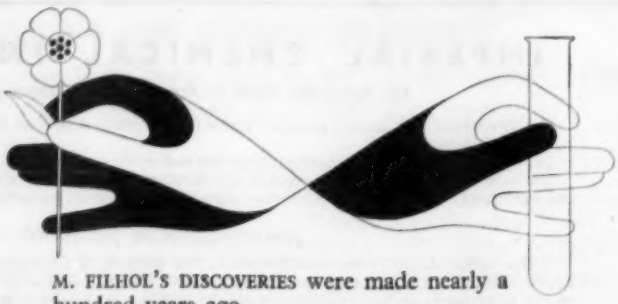
There are two classes of awards —

(*a*) Scholarships which are recognised by the Ministry of Education as a qualification under their regulations for State Scholarships. Awards will be made by the respective Universities in September 1954 on the results of the examination for the General Certificate of Education.

(*b*) Scholarships which may be awarded by the respective Universities to candidates who do not qualify for an award under (*a*) above, but who are considered to be of sufficient merit by virtue of attainments other than the General Certificate of Education. These Scholarships will not qualify the holders for State Scholarships from the Ministry of Education, but will be tenable concurrently with other scholarships.

Candidates should submit their applications to the Registrar of the University of Leeds or the Manchester College of Technology, not later than the 31st July 1954, giving full particulars of their qualifications.

An examination by M. Filhol



"Vegetable Colouring Matters:—M. Filhol has been engaged in the examination of vegetable colouring matters, and has discovered some facts which he now publishes as briefly as possible, intending to give all the details in a longer memoir. There exists in nearly all flowers, says M. Filhol, a substance which is scarcely coloured when in solution in acid liquids, but which becomes of a beautiful yellow colour when acted upon by alkalis." Chemical News, 1860 (April 14) 1 228).

M. FILHOL'S DISCOVERIES were made nearly a hundred years ago.

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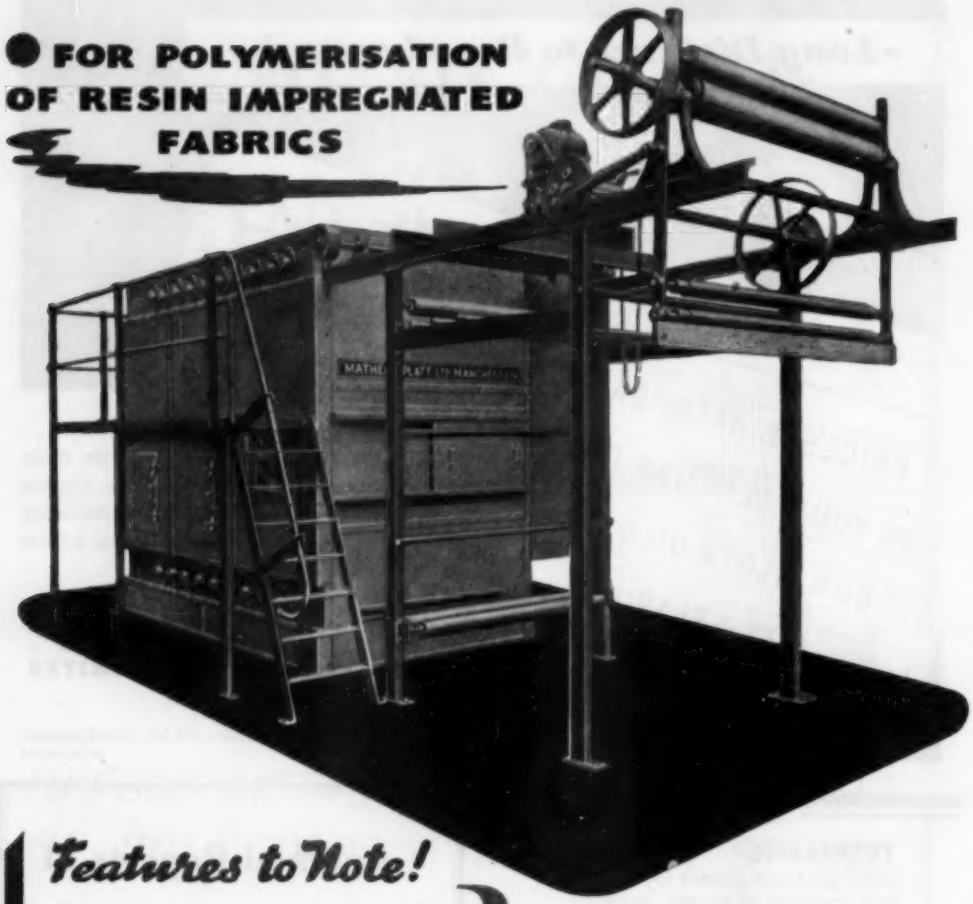

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Review of Textile Progress

Volume 4

Compiled and published jointly by the Textile Institute
and the Society of Dyers and Colourists

Editors

W J HALL A.R.C.S B.Sc
C J W HOOPER Ph.D D.I.C A.R.C.S
Miss C E M JONES M.A

The Textile Institute and the Society of Dyers and Colourists have jointly published a Review of Textile Progress, Volume 4 relating particularly to the year 1952. There are 29 contributors in the Review and the authors are experts in the various sections for which they are responsible

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Manuscripts submitted for publication in the *Journal* should be typewritten (double spacing) on good-quality paper, using one side of the paper only and leaving a margin at least 1 in. wide on the left-hand side. The time taken in refereeing papers (both lectures and communications) will be reduced to a minimum when authors submit two copies of the typescript.

In view of the high costs of publication, it is essential that authors should be as concise as possible. When experimental procedure has already been published, a literature reference to the paper containing the details is sufficient, whilst well known experimental methods should be described very briefly.

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